POTASSIUM CORROSION TEST LOOP DEVELOPMENT

Quarterly Progress Report No. 6
For Quarter Ending January 15, 1965

EDITED BY E. E. HOFFMAN

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION

GENERAL ⊛ ELECTRIC

CINCINNATI, OHIO 45215

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POTASSIUM CORROSION TEST LOOP DEVELOPMENT

QUARTERLY PROGRESS REPORT 6

Covering the Period

October 15, 1964 to January 15, 1965

Edited by

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Lewis Research Center

Under Contract NAS 3-2547

March 12, 1965

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POTASSIUM CORROSION TEST LOOP DEVELOPMENT

I INTRODUCTION

This report covers the period, from October 15, 1964 to January 15, 1965, of a program to develop a Prototype Corrosion Test Loop for the evaluation of refractory alloys in boiling and condensing potassium environments which simulate projected space electric power systems. The envisioned prototype test consists of a two-loop Cb-1Zr facility; sodium will be heated by direct resistance in the primary loop and will be used in a heat exchanger to boil potassium in the secondary, corrosion test loop. Heat rejection for condensation in the secondary loop will be accomplished by radiation in a high-vacuum environment. The immediate corrosion test design conditions are shown below; it is expected that the temperature could be increased by about 400°F when testing is extended to include refractory alloys stronger than Cb-1Zr.

1. Boiling temperature, 1900°F

2. Superheat temperature, 2000°F

3. Condensing temperature, 1350°F

4. Subcooling temperature, 800°F

5. Mass flow rate, 20 to 40 lb/hr

6. Vapor velocity, 100 to 150 ft/sec

7. Average heat flux in the potassium boiler - 50,000 to 100,000 BTU/hr ft^2

The development program is proceeding with the construction and operation of three Cb-1Zr test loops, each of which will be used in a sequence of component evaluation and endurance testing. Loop I, a natural convection loop, has been operated for 1,000 hours with liquid sodium at a maximum temperature of 2260° to 2380°F to evaluate the electrical power vacuum feedthroughs, thermocouples, the method of attaching the electrodes, the electrical resistivity characteristics of the heater segment, and the use of thermal and electrical insulation. Loop II, a single-phase sodium, forced-circulation loop to evaluate the primary loop EM pump, a flowmeter, flow control and isolation valves, and pressure transducers has completed 2,650 hours of scheduled testing. This loop operated at a pump inlet temperature of 1985°F. Residual sodium has been distilled from the loop and component evaluation initiated. The Prototype Corrosion Test Loop, a two-loop system, which has been designed and partially fabricated, will include a boiler, turbine simulator, and condenser in addition to the above components. This facility will be used to develop and endurance test (2,500 hours) the components required to achieved stable operation at the corrosion test design conditions.

The quarterly reports issued for this program will summarize the status of the work with respect to design considerations, construction procedures, and test results. The topical report on Loop I is expected to be released in April, 1965. Detailed topical reports will also be issued to describe each test loop. Additional topical reports will be prepared to cover such areas as materials specification, purification of potassium and sodium, and inert gas purification and analysis.

II SUMMARY

During the sixth quarter, efforts continued on the various phases of the program which is directed at the development and testing of an alkali metal corrosion test system. A necessary and substantial portion of the program effort is the evaluation of the reliability and performance characteristics of both commercially available components and components which have been developed to satisfy the specific needs of the program.

The operation of Component Evaluation Test Loop II, a single-phase sodium, forced-circulation loop to evaluate the EM pump, valves, pressure transducers, and other components which are being used in the Prototype Loop system, completed 2,650 hours of scheduled operation in December, 1964. The highlight of this experiment was the operation of the Cb-1Zr alloy helical induction pump at approximately 2000°F for the 2,650-hour test period.

Fabrication of the Prototype Corrosion Loop progressed substantially during the last quarter. A significant improvement in loop operation characteristics will be realized as a result of the substitution of an iron titanate coated condenser fin in place of a grit blasted condenser fin which has a substantially lower total hemispherical emittance. This change will result in an increase of the heat rejection capability of the condenser by a factor of approximately 1-1/2. Prior to the selection of the high emittance iron titanate coating for the condenser fin, a 1,000-hour test was conducted to evaluate the ability of the coated plate specimens of Cb-1Zr alloy to withstand thermal gradients and thermal cycles more severe than those anticipated during loop operation.

The two refluxing potassium capsule tests which are being conducted to determine the extent of mass transfer of Mo-TZM alloy in Cb-1Zr alloy capsules have completed 1,243 hours of testing at 2000°F. The results of these ancillary experiments will contribute to an understanding of corrosion phenomena which may be encountered in the turbine simulator of the Prototype Corrosion Loop.

III PROGRAM STATUS

A. Component Evaluation Test Loop II

1. Loop II Operation

During the quarter Loop II was operated an additional 615 hours and completed the scheduled 2,500-hour test at 1000 hours on November 6, 1964, at the following test conditions:

Heater outlet temperature	-	2050°F
Pump inlet temperature	-	1985°F
Metering valve temperature	-	650° - 800°F
Loop pressure	-	144 psia
Sodium flow rate	-	1 gpm (400 lb/hr)
Sodium velocity (1/4-inch ID)	-	7 fps
Electric power input	-	6.75 KW
Vacuum chamber pressure	-	8×10^{-9} torr

The operation history of the 2,500-hour Loop II test and the 150 hours of post-test operation is given in Table I. After a series (6) of interruptions during the first 1,154 hours, which were reported last quarter[1] in detail, Loop II operated without interruption for the remaining 1,346 hours. The temperatures of the various regions of the loop during the test are given in Figure 1.

The pressure rise of the Loop II chamber was obtained with the loop at temperature (2000°F) and following cooling of the loop and the chamber to room temperature. Results of these are given in Table II and indicate that for both the hot and cold tests the pressure rise rate was higher in the period 0 to 10 minutes than from the period 10 to 45 minutes. This high initial rise rate is attributed to the release of inert gas from the getter-ion pump elements. The low pressure rise rates for both tests indicate that the system was extremely tight and clean. The indicated ionization gauge pressure and the partial pressures of the various gaseous species for the Loop II test chamber environment during 2,500 hours of test operation are given in Figure 2.

The first 50 hours of the test is shown on an expanded time scale to illustrate the rapid changes in total and partial pressures during this period. No attempt has been made to interpret each of the pressure fluctuations

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[1] _Potassium Corrosion Test Loop Development_, Quarterly Progress Report No. 5 for Period Ending October 15, 1964, NASA Contract NAS 3-2547, NASA-CR-54269.

TABLE I

LOOP II - OPERATION HISTORY

July 2 - December 2, 1964

Time	Date	Test Hours	
1000	July 2	0	Loop start-up
1130	July 19	409	Unscheduled shutdown - overtemperature of EM pump thermal overload relay
1330	July 20		Resume test
0800	August 3	734	Scheduled shutdown - metering valve modified by substituting L-605 bushing and stem for original parts - chamber wall deposits analyzed - thermocouples repaired
	August 18	734	Attempt to resume test - metering valve and flow difficulties
	August 18	734	Scheduled shutdown - Saginaw ball nut and stem substituted for L-605 stem and stem bushing in metering valve
1300	August 22		Resume test
0610	August 23	757	Unscheduled shutdown - defective ion-pump cell
1300	August 24		Resume test
1930	August 24	763	Unscheduled shutdown - argon instability in ion pump
1430	August 25		Resume test
0900	September 10	1154	Unscheduled shutdown - argon instability in ion pump
2000	September 10		Resume test
1000	November 6	2500	Scheduled test completed
0900	November 24		Start of post-test operation
1600	November 27	2600	End of scheduled 100-hour run
1100	November 30		Start of 50-hour flow instability study
1030	December 2	2650	End of Loop II operation

Vacuum Tank

Pressure Transducers

Flowmeter

Pump Outlet

EM Pump

Pump Inlet

Gas Pressurization Line

Drain and Fill Line

Resistance Heated Coils

Electrode

Valves

Valve Bypass

Surge Tank
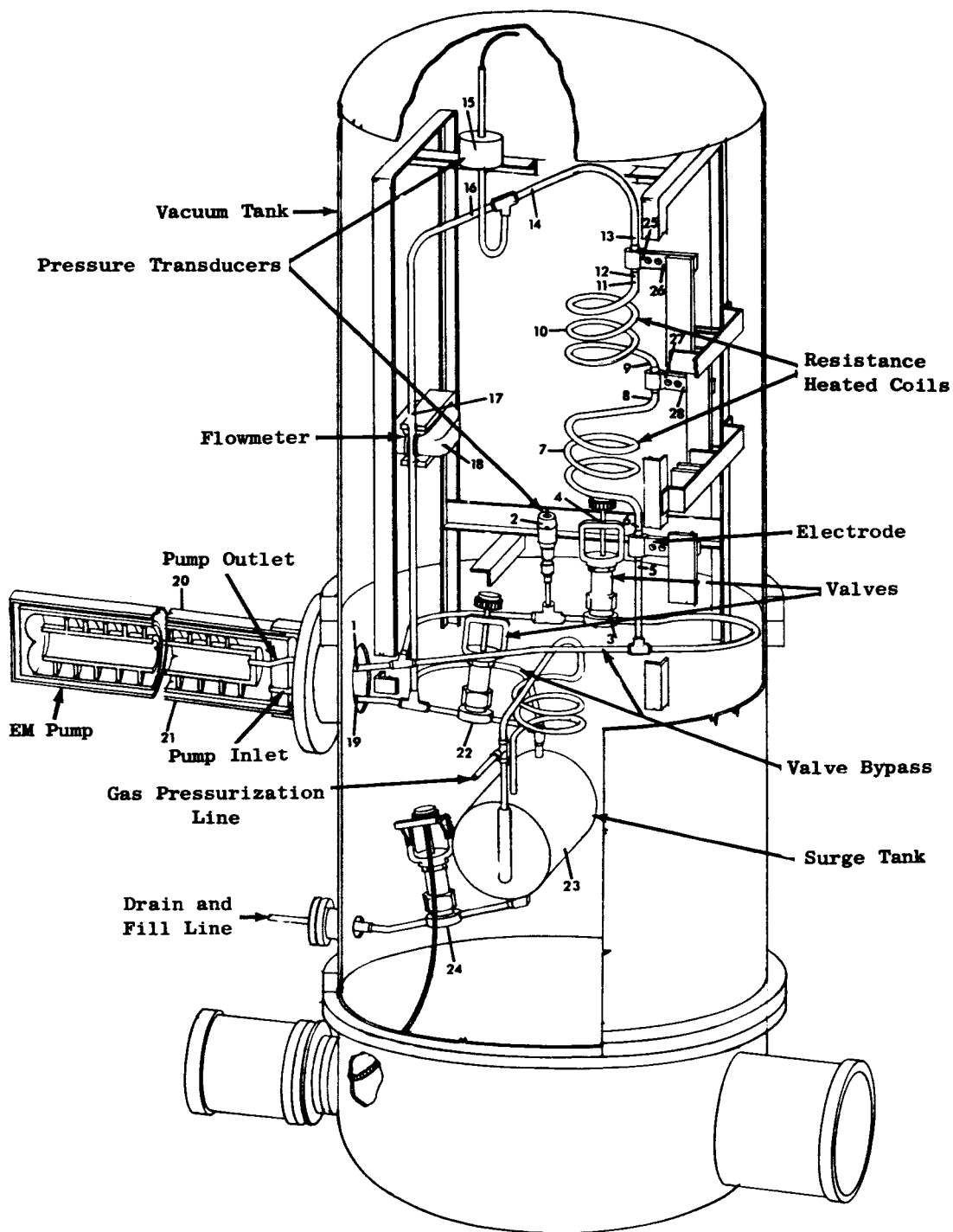
Loop Temperatures	
T/C No.	°F
1	1975
2	585
3	800
4	580
5	1965
6	1950
7	1985
8	2020
9	2000
10	2035
11	2065
12	2065
13	2050
14	2045
15	650
16	2030
17	2015
18	670
19	1985
20	1880
21	1850
22	505
23	475
24	455
25	1655
26	1575
27	1720
28	1580

Figure 1. Component Evaluation Test Loop II Showing Location of W-3%Re vs W-25%Re Thermocouples.

TABLE II

PRESSURE RISE OBSERVED ON LOOP II TEST CHAMBER

Time, Minutes	Loop at Temperature (2000°F) Pressure, Torr	Loop and Chamber at Room Temperature Pressure, Torr
0 (Getter Ion Pump Off)	1.9×10^{-8}	8.2×10^{-10}
1	1.3×10^{-7}	6.5×10^{-9}
2	2.3×10^{-7}	1.5×10^{-8}
3	2.8×10^{-7}	2.3×10^{-8}
4	3.3×10^{-7}	3.1×10^{-8}
5	3.7×10^{-7}	3.8×10^{-8}
6	4.0×10^{-7}	4.5×10^{-8}
7	4.2×10^{-7}	5.2×10^{-8}
8	4.4×10^{-7}	5.8×10^{-8}
9	4.4×10^{-7}	6.5×10^{-8}
10	4.5×10^{-7}	7.1×10^{-8}
15	4.8×10^{-7}	9.9×10^{-8}
20	4.8×10^{-7}	1.2×10^{-7}
25	4.8×10^{-7}	1.4×10^{-7}
30	4.8×10^{-7}	1.6×10^{-7}
35	4.9×10^{-7}	1.8×10^{-7}
40	4.9×10^{-7}	1.9×10^{-7}
45	5.0×10^{-7}	2.0×10^{-7}

Pressure Rise Rate Observed for Time Period 10 to 45 Minutes Above:

Loop at Temperature: 1.43×10^{-6} Microns/Minute
(Time Required for Pressure to Rise One Micron: 486 Days)

Loop and Chamber at Room Temperature: 3.7×10^{-6} Microns/Minute
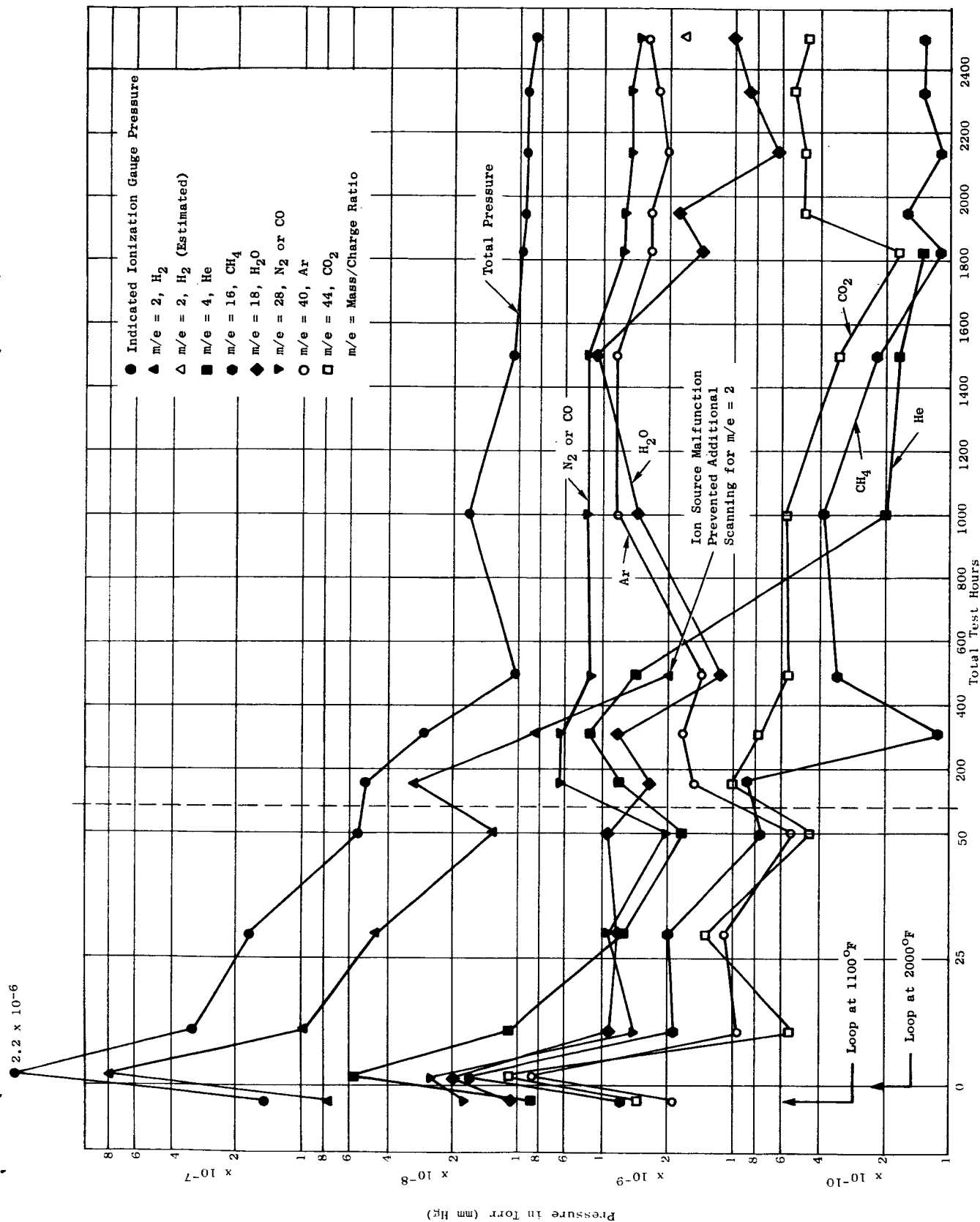(Time Required for Pressure to Rise One Micron: 188 Days)

Figure 2. Indicated Ionization Gauge Pressure and Partial Pressures of the Various Gaseous Species in the Loop II Test Chamber vs Total Hours of Loop Operation.

of the individual species, but only to characterize the general trends of the principal gases. It may be noted that during the first 200 hours of the test, the hydrogen partial pressure was approximately 10 times higher than any other gas in the system. Unfortunately, the hydrogen peak was lost after 500 hours of test operation due to malfunction of the ion source at high accelerating voltages. Comparison of the sum of the partial pressures and the total pressure as indicated by the ion gauge before and after the loss of the hydrogen peak indicates that the hydrogen pressure was $1-2 \times 10^{-9}$ torr during the last 2,000 hours of the test. It is of interest to note that the ionization gauge pressure continues to drop at a rate of approximately 0.2×10^{-10} torr/100 hours even after 2,000 hours of loop operation.

2. Post-Test Component Inspection

The visual inspection of the loop after test showed that 3 of the 6 copper electrical leads to the stressed diaphragm pressure transducer were open about 4 inches from the LVDT connection. The failure was probably due to mechanical fatigue caused by induced vibration from the heater power supply. A generous thermal expansion loop in the copper wire leads had been provided to accommodate the differential thermal growth between the support structure and the loop and permitted large amplitude swings.

A check of the metering valve actuation system showed that the operating difficulty during the test was due to galling of the stainless steel gear shaft in the stainless steel support bracket attached to the valve. The assembled valve including the gear shaft and support bracket were illustrated in an earlier progress report[2]. This problem had been observed at an earlier inspection and an attempt to alleviate the problem by increasing the bearing surface of the shaft was not successful. A new bracket and shaft have been designed for the Prototype Loop with a tungsten carbide bushing to eliminate this problem.

At the completion of the 2,500-hour test, 13 of the 28 thermocouples had failed. The post-test examination of the 13 failed thermocouples showed that 5 thermocouples were broken at the vacuum feedthrough, one thermocouple was open in the reference junction and 7 thermocouples were shorted at the vacuum feedthrough. Thermocouple failures at the vacuum feedthrough were a major cause of thermocouple difficulty in Loop I and Loop II due to the poor bend ductility of the W-3%Re wire.

A radiographic inspection of the metering valve and loop tees was not successful in locating any high-density corrosion or mass transfer particulate matter which would explain the difficulty in maintaining flow in the bypass line. A radiograph of the vapor trap on the argon pressurization line showed no detectable accumulation of sodium indicating that the addition of the condensing coil eliminated the collection of sodium in the gas line as was observed in Loop I during assembly.

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[2] *Potassium Corrosion Test Loop Development*, Quarterly Progress Report No. 4 for Period Ending July 15, 1964, NASA Contract NAS 3-2547, NASA-CR-54167, p 36.

3. Additional Test Operation

In addition to the completion of the 2,500-hour endurance run, Loop II was operated at the design test conditions for an additional 100 hours to evaluate:

1. A new gear assembly for the metering valve.

2. Relocation of the thermocouple reference junction block inside the vacuum chamber.

3. A new type of high vacuum thermocouple feedthrough with W-3%Re and W-25%Re thermocouple wires with ceramic-to-metal seals replacing the nickel feedthrough tubes used to-date.

4. Replacement of the copper lead wires on the stressed diaphragm pressure transducer with stainless steel lead wires.

5. A 15 KW Hall effect wattmeter used to measure accurately the electric power input to the sodium heater.

Although the loop was filled (from the surge tank) and flow was established on November 23 without difficulty, large flunctuations in both the flow rate and loop pressure were observed before power was applied to the loop. The flunctuations increased in amplitude as the loop temperature was increased and continued during the entire 100-hour test.

During the test, repeated operation of the metering valve showed that the new gear assembly was a significant improvement over the previous design. The new gear bracket not only provided a more accurate alignment of the gears but permitted the installation of a tungsten carbide bushing that eliminated the galling problem encountered in the 2,500-hour endurance run. The new design will be used in both the on-off and the metering valves of the Prototype Loop.

The relocation of the thermocouple reference junction block from a constant temperature (150°F) reference junction outside of the vacuum chamber to a reference junction inside of the chamber was made to eliminate the problems which have been encountered in obtaining helium leak tight brazed joints between the W-Re thermocouple wires and the nickel tubes of the high-vacuum feedthroughs. Failure of the W-3%Re wire near the braze joint during the fabrication and installation of the thermocouples was responsible for a large number of thermocouple failures in both the Loop I and Loop II tests.

The new reference junction block consists of a high-purity alumina terminal strip mounted on a 1/4-inch copper plate attached to the chamber wall. Tantalum foil thermal shields were used to reduce the radiation heat flux from the test loop and to maintain the junction block at a temperature of less than 200°F and as isothermal as possible during loop operation. The temperature of the reference junction which is required to correct the indicated loop temperature is measured by three copper-constantan thermocouples.

The relocation of the reference junction inside the chamber and the use of the brazed copper leads eliminated a major portion of the thermocouple failure troubles experienced in the past. The copper wire leads were more ductile and the routing of the wires both inside and outside of the vacuum chamber was facilitated.. Not all of the braze joints between the copper wires and the nickel tubes of the vacuum feedthrough were helium leak tight following the first brazing operation in the argon-filled chamber; however, the problem encountered is far simpler to solve than the brazing of W-Re wires into the nickel tubulations. This is due primarily to superior brazing characteristics of the copper wire/nickel tube combination compared to the W-Re wire/nickel tube combination.

The new type of thermocouple feedthrough, which consists of W-3%Re and W-25%Re wire brazed with a copper-gold alloy into an alumina disk, was evaluated in the 100-hour run following completion of the scheduled 2,500-hour test, is shown in Figure 3. This feedthrough* has W-Re thermocouple wire penetrations in lieu of the standard nickel tubulations to minimize the possibility of spurious thermal emf's generated from dissimilar metal contacts. No difficulties were experienced in the installation and the seals proved reliable and remained leak free during the 500°F bakeout and the subsequent loop operation. The use of this type of feedthrough requires the use of an external thermocouple junction.

A 15 KW Hall effect wattmeter** was installed in the heater power supply of Loop II after an initial calibration at the factory. The wattmeter has a 0-1.5 KW, 0-4.5 KW and 0-15 KW range selector switch with a ± 1% accuracy. The input power calculated from the potential and current measured at the primary to the 20 KVA transformer was approximately 10% higher than the output power measured with the wattmeter. However, when the transformer core losses were accounted for, the agreement was within 5%. The wattmeter will be used for accurate power measurements in calibrating the primary and secondary flowmeters of the Prototype Corrosion Loop by calorimetric measurements. Although precise power measurements were not required for Loop II operation, the installation of the wattmeter at this time was made to checkout the equipment and familiarize program personnel with the operation of this unit before the Prototype test.

Upon completion of the 100-hour period of post-test operation, the sodium was allowed to freeze in the loop and radiographic examination of the loop revealed spherical shaped void areas in both the vertical and horizontal pipe sections of the loop. These voids are believed to be accumulations of argon gas which entered the flow circuit via the surge tank and dip leg.

The test was continued for an additional 50-hour period to study the frequency and magnitude of the instability in the loop due to the apparent gas accumulation and possible corrective action which would be effective in future loop operations. The unstable operation could be observed and measured not only on the flow rate recording but similar perturbations were

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* General Electric Vacuum Products Operation, Schenectady, New York, (Model 22HN020).

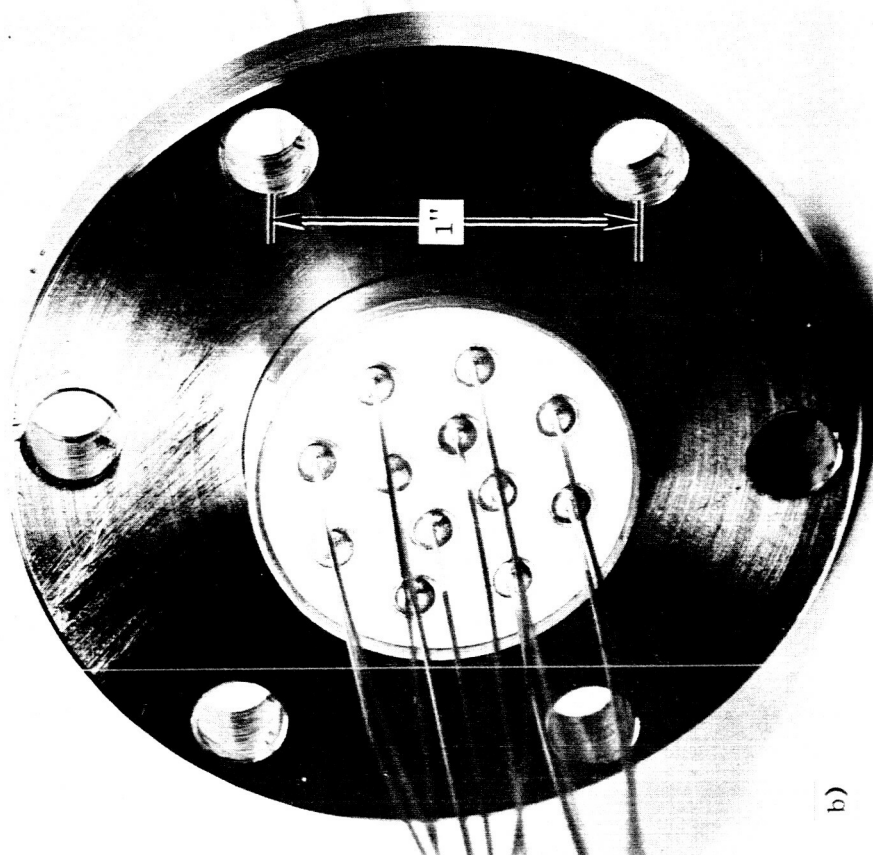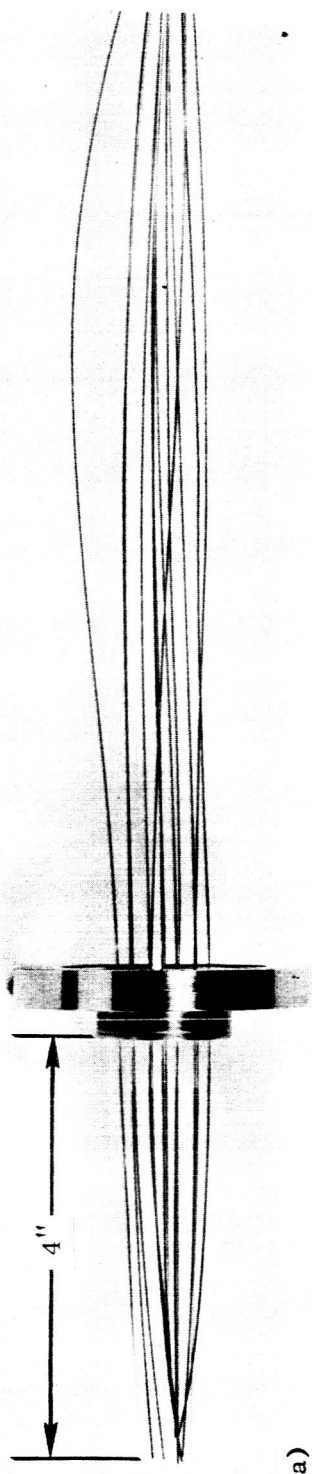** Columbus Scientific, Inc., Columbus, Ohio, (Model 150C61-YM).

Figure 3. Thermocouple Feedthrough Flange with 0.020-Inch W-3%Re and W-25%Re Thermocouple Wires Brazed in Ceramic Insulator. a. Over-all View of Feedthrough (C6411837). b. Closeup View of Feedthrough (C64-111836).

recorded for the heater temperature and loop pressure. The maximum tempera-
ture fluctuations during the test were approximately ± 25°F with an average
frequency of 6 cycles per second. The loop operation could be stabilized
by reversing the flow. It is possible that the improved stability was due
to a more favorable geometry at the pump inlet for trapping gas in the pump
cavity when the flow was reversed.

In order to attempt to remove the argon from the flow circuit, the
sodium was dumped from the loop to the surge tank by evacuating the surge
tank through the pressurization and evacuation line. The sodium was held in
the surge tank under vacuum for several hours at 450°F and then pressurized
back into the loop. No flow or temperature flunctuations were detectable when
the loop was restored to the test conditions with both normal and reversed
flow indicating that the procedure outlined above had removed the argon from
the flow circuit.

As a result of these experiments and a review of the previous occa-
sions when flow instabilities were observed, it has been concluded that the
argon was introduced into the loop during the filling operation. The standard
procedure used to fill the loop was to pressurize the surge tank to 50 psia
and then melt the sodium in the surge tank by turning on the chamber bakeout
heaters. It now appears that localized melting of the sodium in the vicinity
of the dip line, which extends to the bottom of the surge tank, could have
allowed both sodium and argon to enter the loop.

The loop filling procedure for the Prototype Loop has been modified
to avoid argon entrainment during the filling of the primary and seoondary
circuits. A vacuum will be maintained above the sodium and potassium in the
surge tanks until the alkali metal is molten. The surge tanks will then be
pressurized with argon to force the alkali metal into the loop.

4. Draining, Sampling, and Distilling of Sodium

Following completion of the 50-hour test, the Loop II sodium was
transferred to the disposal tank outside the test chamber. A sodium sample
was taken as the loop was dumped.

A 5-micron Type 316 stainless steel filter, 3/4-inch diameter by
1/8-inch thick, had been installed in the dump line from Loop II in order to
collect any particulate matter which might be present in the sodium.

It was planned to filter the Loop II sodium with a pressure drop
across the filter of 13-15 psi to avoid breaking or blowing out the filter.
However, the loop dumped suddenly at about 1000°F when the sodium in one of
the transfer lines melted. It was determined that a by-pass valve around the
filter was partially open and very little sodium passed through the filter.
The analyses of the sodium sample obtained during the dump are in Table III
and indicate oxygen at the 5 ppm level and a very low concentration of other
elements. Although only a portion of the sodium went through the 5-micron
stainless steel filter when the loop was emptied, the filter assembly was
removed and vacuum distilled until free of residual sodium. Examination of

TABLE III

ANALYSIS OF POTASSIUM OBTAINED FROM LOOP II

AFTER 2,650 HOURS OF OPERATION

Oxygen Analysis

	Sample Wgt. gms	ppm O as Na_2O By Mercury Amalgamation Method
Sample No. 149-192	4.543	4.9
	2.180	5.8

Spectrographic Analysis

Element	GE ppm of NaCl	NUMEC ppm of NaCl
Fe	<5	3
Al	1 or less	1
Ag	<1	<1
B	<25	<5
Be	<1	<1
Ca	1-15	4
Cb	<1	<10
Co	<1	<5
Cr	<1	<1
Mg	<1	<1
Mn	<1	<1
Mo	<1	<5
Ni	<1	<1
K	--	60
Si	<1	<10
Sn	<1	<5
Ti	<1	<5
V	<25	<1
Zr	<1	<10
Pb	<1	<5
Cu	<1	<1

NUMEC = Nuclear Materials & Equipment
        Corporation, Apollo, Pennsylvania

the filter at a magnification of 30X revealed no mass transfer particles and spectrographic analysis of metal scraped from the face of the filter showed only the normal stainless steel elements.

Following the dumping of the sodium from the loop, the test chamber was opened and modifications were made in order to distill residual sodium from the loop. Six tantalum coil heaters with a total heat capacity of 2 KW were installed on the loop support structure and a stainless steel thermal radiation shield was added between the loop heaters and the vacuum chamber wall to reduce the heat losses. The chamber was resealed and evacuated to the 10^{-7} torr range. The tantalum heaters and the chamber bakeout heaters were then turned on and the power input was increased slowly for several hours until the minimum loop temperature was 800°F. Power was also supplied to the heater coil and the EM pump and their temperatures were maintained at 1000°F. The distillation was continued for over 200 hours during which time the sodium was periodically drained from a small collection tank into the disposal tank.

In order to establish if any particulate matter from Loop II had been missed in the dumping procedure outlined above, the sodium in the disposal tank was filtered during transfer from the disposal tank to another storage container. The 5-micron stainless steel filter assembly used was removed, distilled free of residual sodium, and subsequently inspected. Several dark areas were detected on the face of the filter. These areas were scraped with a metal spatula, and the particles were removed and analyzed. Only the constituents of stainless were recovered. Sodium samples for additional oxygen determinations were collected during the transfer from the disposal tank to the storage container. Duplicate analyses on this sample produced oxygen values of 5.3 and 7.6 ppm which are in good agreement with the values of 4.9 and 5.8 ppm given in Table III. No significant concentration of columbium, zirconium or other metallic elements was detected in the analysis of a spectrographic sample. It is tentatively concluded that complex sodium-columbium-oxygen compounds were not responsible for the occasional flow difficulties experienced during Loop II operation.

During the next quarter the loop will be disassembled. Metallographic and chemical analysis will be performed on specimens from the various regions of the loop.

5. Loop II Stressed Diaphragm Pressure Transducer

As reported above three of the six copper electrical leads connecting the stressed diaphragm pressure transducer LVDT coil to a terminal strip were broken during the 2,500-hour test. These wires were replaced with Type 302 stainless steel wires for the post-test operation to increase the mechanical strength. This also eliminates any possible thermoelectric potential due to dissimilar metal junctions between the lead wire and the Type 302 stainless steel header pin of the coil.

During the pump down of the vacuum chamber before restoring the loop to the test conditions, the pressure transducer behaved normally during the roughing operation when sorbtion pumps were used to evacuate the chamber to approximately 5 microns. When the ion pump was turned on, the transducer output became very unstable and could be observed to be associated with the electrical arcing in the chamber prior to confinement of the ion pump. Upon confinement of the ion pump, the transducer output again became stable although the zero level was displaced from the original zero by approximately 200 millivolts. The transducer output in response to a change in pressure level remained normal and showed a 60 millivolt change for a 127 psi change in pressure during a subsequent calibration.

During the pump down of the vacuum chamber for the distillation of the residual sodium from Loop II after completion of 2,650-hour test, it was observed that 300 volt D.C. potential appeared on several non-grounded conductors during the ion pump startup. The effect of this D.C. voltage on the electronic circuitry may be responsible for the erratic behavior and zero shift observed during the 2,500-hour test. If the zero shift is due to a permanent change in the electrical characteristics of the excitation circuitry incurred during the ion pump starting period, this problem can be eliminated by disconnecting the electronic circuitry during the pump down cycle in future tests.

Preliminary calibration tests at room temperature using argon gas have indicated a net output change of 40 millivolts for a 150 psi change in pressure with a sensitivity of 6 psi per millivolt at the 150 psig level. Zero stability and temperature sensitivity tests will be made during the reporting period.

These tests were delayed during the post-test operation of Loop II because the same excitation-demodulation circuit is used in both tests. Following the post-test operation, the excitation demodulation circuit was again connected to the second transducer, and a negative zero shift of 170 millivolts was observed. Since no change in the transducer had occurred during the test delay, it must be assumed that the zero shift was caused by a change in the electronic circuitry and probably due to the high voltage arcing observed during the ion pump startup prior to the post-test operation of the loop. As mentioned above, this problem will be eliminated in Prototype Corrosion Loop operation by disconnecting the system during the chamber pump down period.

B.   Prototype Corrosion Loop Design

Several changes have been incorporated into the loop design during the last quarter and the final version of the system is illustated in Figure 4.

A subcooler reservoir has been added between the condenser and EM pump inlet of the secondary potassium circuit to increase the available liquid supply to the pump during test operation. During periods of instability,
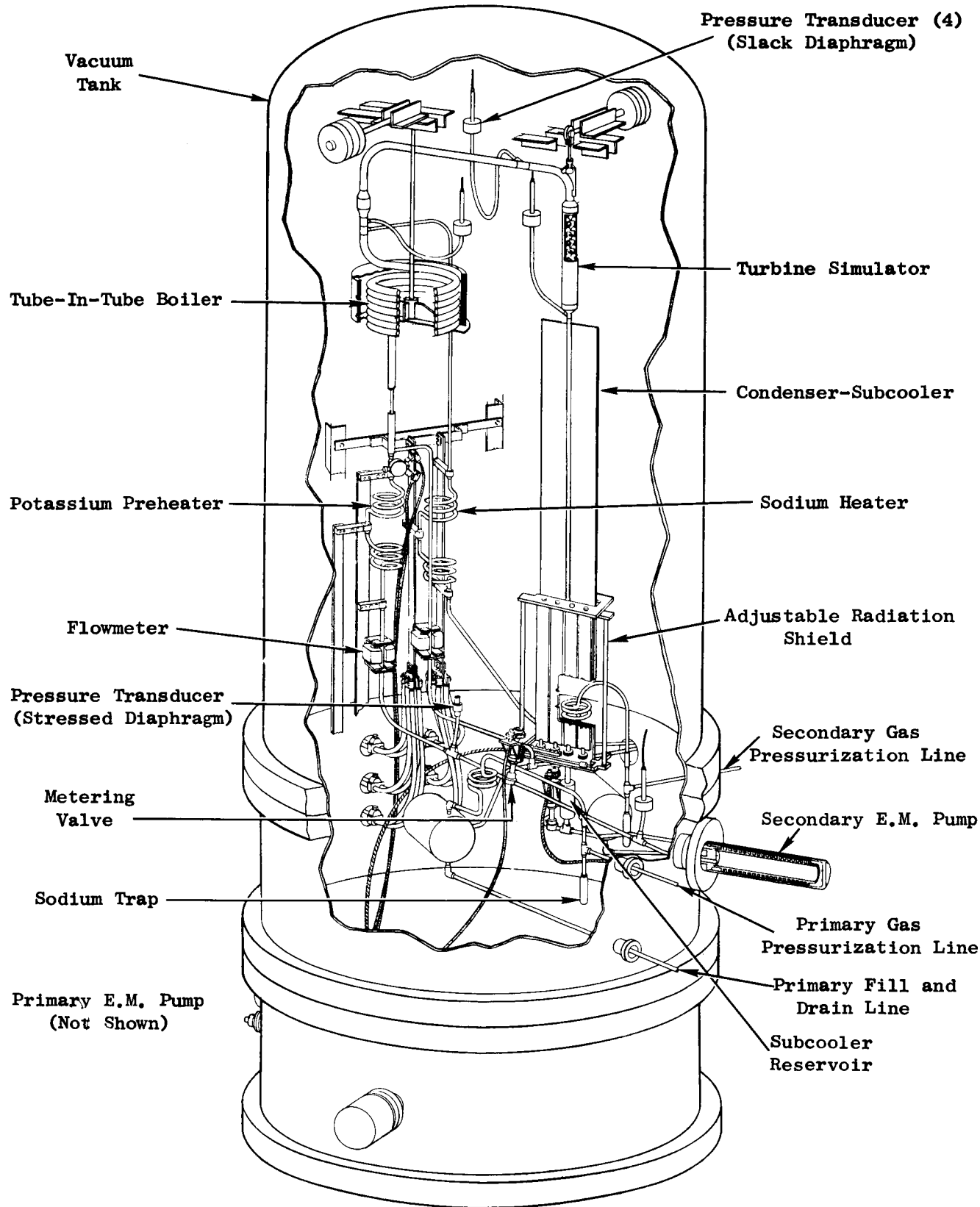
Figure 4. Isometric of the Prototype Corrosion Loop.

Vacuum Tank

Pressure Transducer (4) (Slack Diaphragm)

Tube-In-Tube Boiler

Turbine Simulator

Condenser-Subcooler

Potassium Preheater

Sodium Heater

Flowmeter

Adjustable Radiation Shield

Pressure Transducer (Stressed Diaphragm)

Secondary Gas Pressurization Line

Metering Valve

Secondary E.M. Pump

Sodium Trap

Primary Gas Pressurization Line

Primary E.M. Pump (Not Shown)

Primary Fill and Drain Line

Subcooler Reservoir

the liquid-vapor interface would be oscillating and could conceivably move into the EM pump inlet, further aggravating flow and pressure fluctuations. The addition of the subcooler reservoir will increase the subcooler "pumpout" time from 8 seconds to 21 seconds at the design flow rate of 40 lbs/hr.

The addition of the subcooler reservoir required that the condenser be shortened from 64 inches to 58 inches because of space limitations. This would result in the liquid-vapor interface being approximately 6 inches from the condenser exit. A high emittance coating (Fe_2TiO_5) will be applied to the condenser fins to increase the fin emittance from 0.4 (grit blasted) to 0.86. The effect of the higher emittance surface will reduce the required condenser length from 52 inches to 37 inches thus raising the liquid-vapor interface from 6 inches to 21 inches from the condenser exit. A thermal cycling test on this coating was conducted and is described in detail later in this report.

The redesign of the condenser shield was completed. The new design includes a ball bearing screw and four ball bearing splines to reduce the galling tendency of rubbing surfaces in vacuum due to self-welding.

The valve gear assembly was also redesigned for an increase in the turning ratio from 2 to 1 to approximately 5 to 1. The higher turning ratio will result in an improvement in the ability of the loop operator to determine the exact position of the metering valve and require less operating torque at the magnetic rotary feedthrough.

C.   Prototype Corrosion Loop Fabrication

1.   Boiler Assembly

The fabrication of the boiler assembly proceeded smoothly through the welding operations which precede the coiling operation. The assembly which consisted of the inner and outer tubes with spacers attached was formed by Philadelphia Pipe Bending Company. Prior to forming, the inner tube and the annulus were packed with sugar to aid in maintaining concentricity. The forming operation was conducted without difficulty.

Upon receipt of the formed boiler from the vendor, the sugar was removed by dissolving and rinsing in filtered hot water. Approximately 12 hours of rinsing were employed, four hours on the inner tube and four hour in each direction on the annular spacing. Distilled water was used for a final rinsing operation. Application of the Molisch Test to samples of the final rinse water indicated no detectable sugar content.

The cleaned boiler assembly was then radiographed to determine the concentricity of the inner and outer tubes. Examination of these radiographs indicated that good concentricity was maintained. Radiographs of the potassium boiler inlet tube and the boiler coils are shown in Figures 5 and 6.
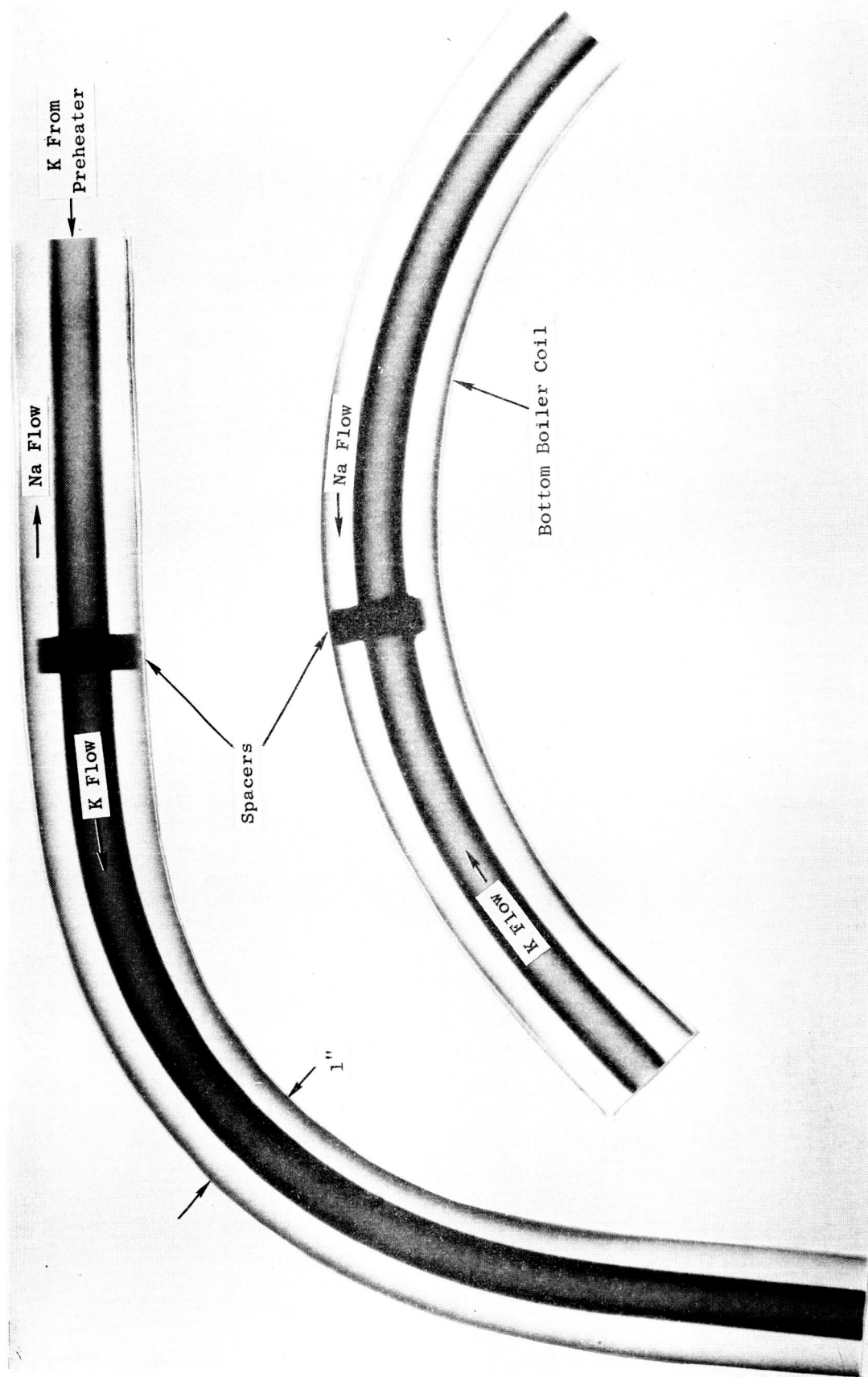
Figure 5. Radiographs of Portions of the Prototype Corrosion Loop Boiler Coil. Packed Sugar was Used to Maintain Concentricity of Tubes During Forming.
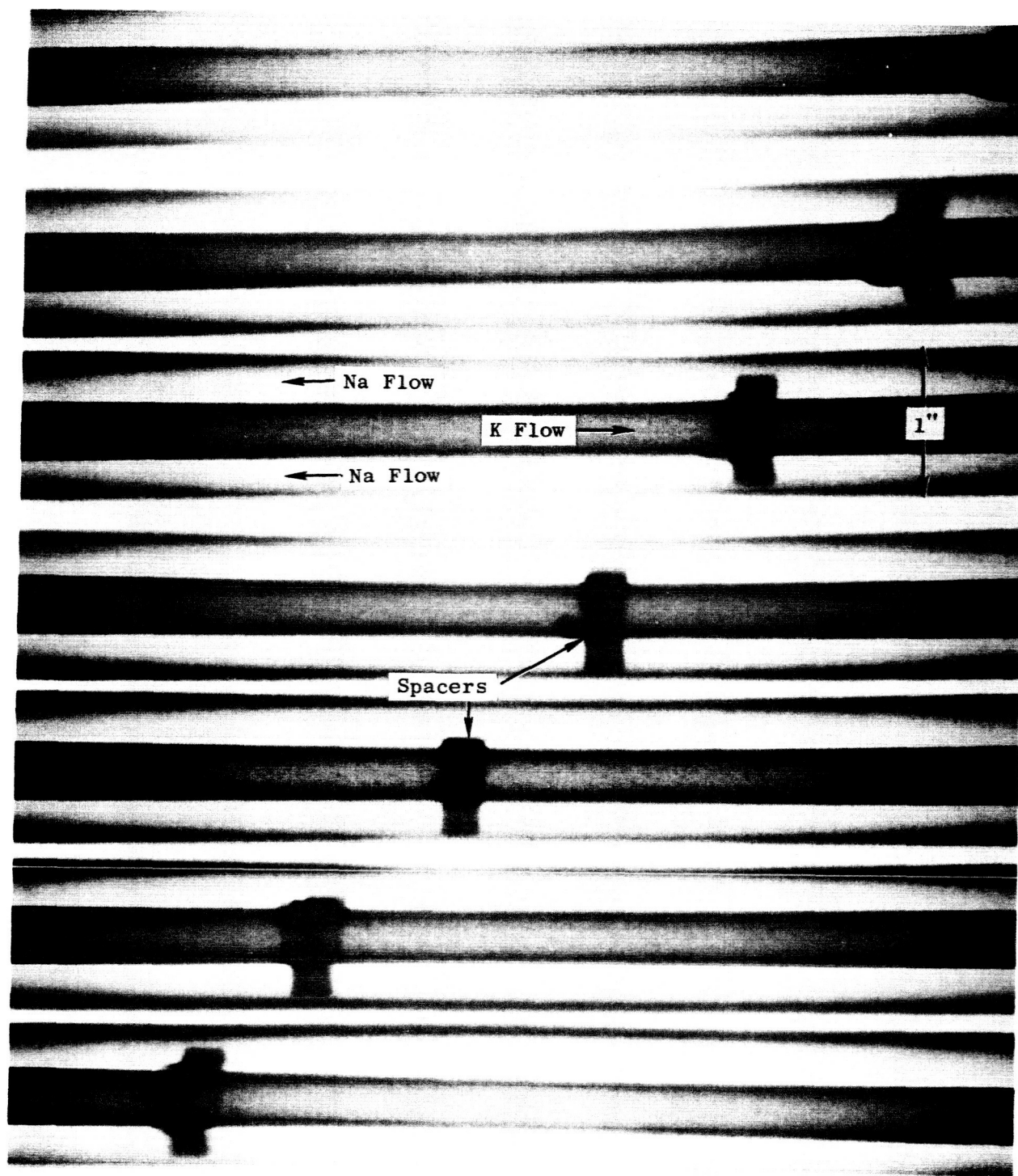
Figure 6. Radiograph of Prototype **Corrosion Loop** Boiler Coils. Packed Sugar **was** Used to Maintain Concentricity of Tubes During Forming.

2. Turbine Simulator

The machining of the turbine simulator nozzle assemblies continued during this period. The Mo-TZM alloy nozzle throats and blade assemblies were electric discharge machined without difficulty. The machining steps required to produce the nozzle configuration are shown in Figure 7.

The final Elo-polish, a refinement of electric spark discharge machining, produced a 32-rms surface finish in the nozzle throat. This surface was further polished with 400-grit alumina paper to remove approximately 0.001 mil from the surface. A final polish with one-micron alumina produced an 8-rms finish. Zyglo inspection of the finished parts indicated that the surfaces were free of cracks.

The Cb-1Zr components for this subassembly have been completed with the exception of the outer casings. These components are scheduled for completion early in the next report period. Upon receipt of these casings, the nozzles will be machined to the final required dimensions required to obtain the slip fit of the nozzle assemblies into the casings.

3. Bimetallic Joints

Four joints between the Cb-1Zr and Type 316 stainless steel process tubes are required to provide alkali metal fill and gas pressure line connections. The machined components of these joints are illustrated in Figure 8. Process tubes were welded to the Cb-1Zr component, and the welds were heat treated prior to brazing of the joints. The brazing was performed in accordance with Specification SPPS-9A.

4. Vapor Nucleator

The vapor nucleator components are shown in Figure 9 before and after welding. The nucleator cavity is located in the "heat lens", which is heated by a tungsten coil, and consists of a stepped hole with the larger entrance cavity leading to a smaller nucleator cavity.

5. Potassium Preheater Coil

The welding of the potassium preheater subassembly was completed. The electrodes are shown in Figure 10 prior to welding. A completed electrode shown in Figure 11 illustrates the welding of the thermocouple welds and the joint between the electrode and electrode bar. The completed subassembly with the heater coils and vapor nucleator welded in place is shown in Figure 12. The heater elements and thermal insulation for the vapor nucleator will be installed during final assembly of the loop in the vacuum chamber.

6. EM Pump Ducts

Fabrication of the primary and secondary EM pump ducts was completed. The shrink fits between the finned ducts and outer wrappers were
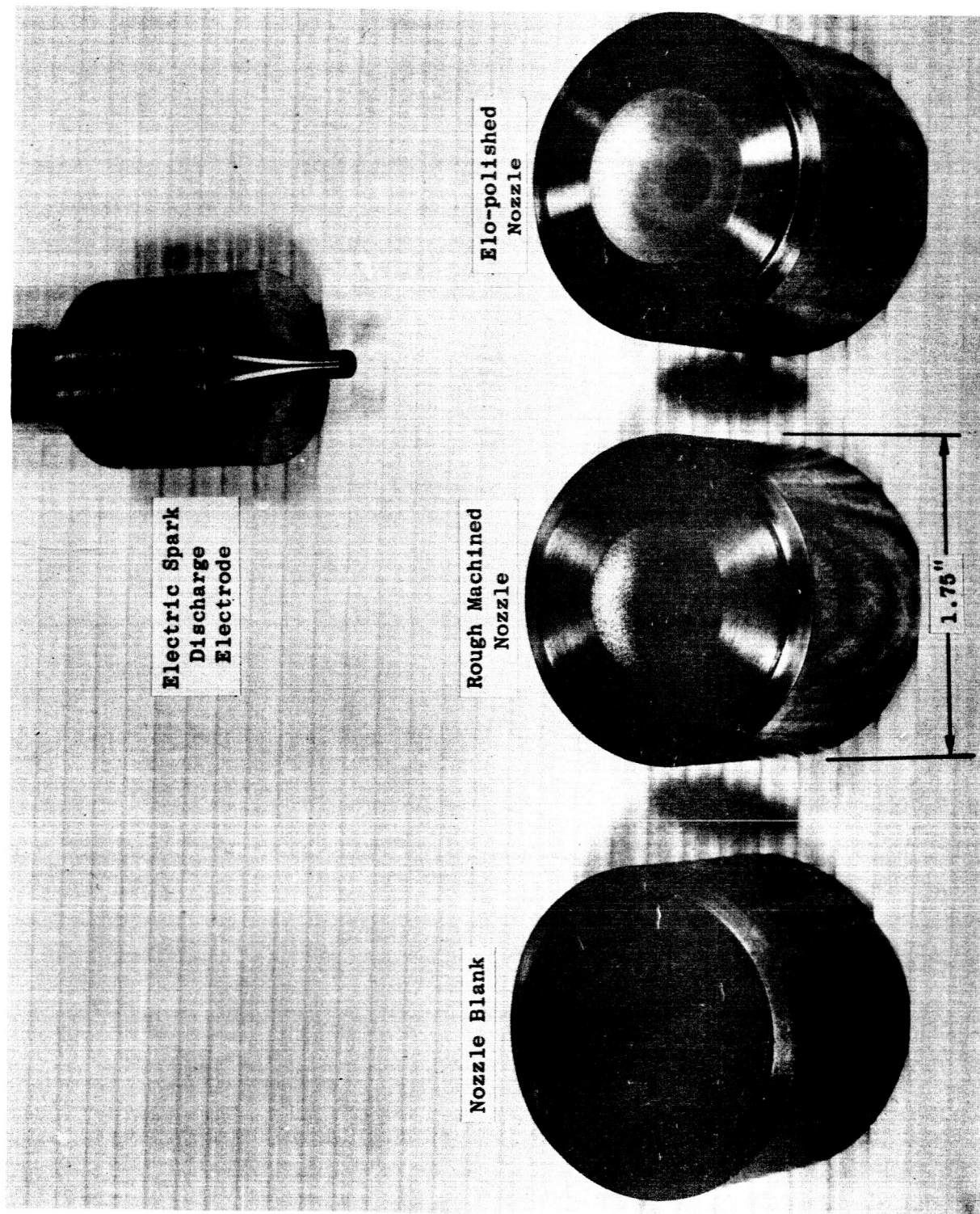
Figure 7. Machining Sequence Required to Produce the Mo-TZM Alloy Nozzle for the Prototype Corrosion Loop Turbine Simulator. Two Additional Polishing Operations are Required to Produce an 8-rms Surface Finish. (Orig. - C6412207)
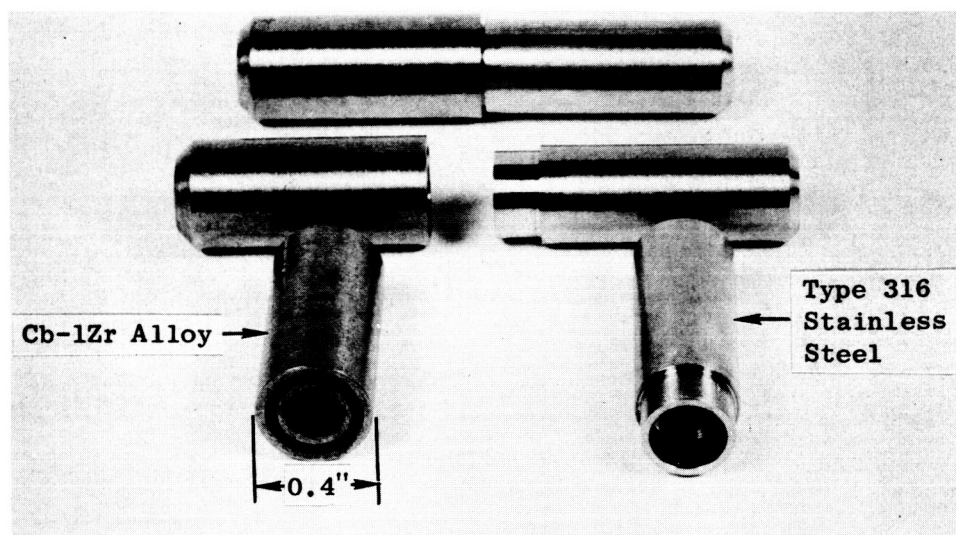
Figure 8. Bimetallic Joint Between Cb-1Zr Alloy and Type 316 Stainless Steel. Process Tubes, 0.375-Inch OD by 0.065-Inch Wall Thickness, are Welded to the Cb-1Zr Alloy Component Prior to Brazing. (Orig. - C64122908)

Radiant Heat from W Element

Thermocouple Well

b.

Nucleator Cavity

$\left\{ \begin{array}{l} \text{0.125" Dia. x 0.25" Deep} \\ \text{0.062" Dia. x 0.63" Deep} \end{array} \right.$

K to Boiler

1"

K from Preheater

a.
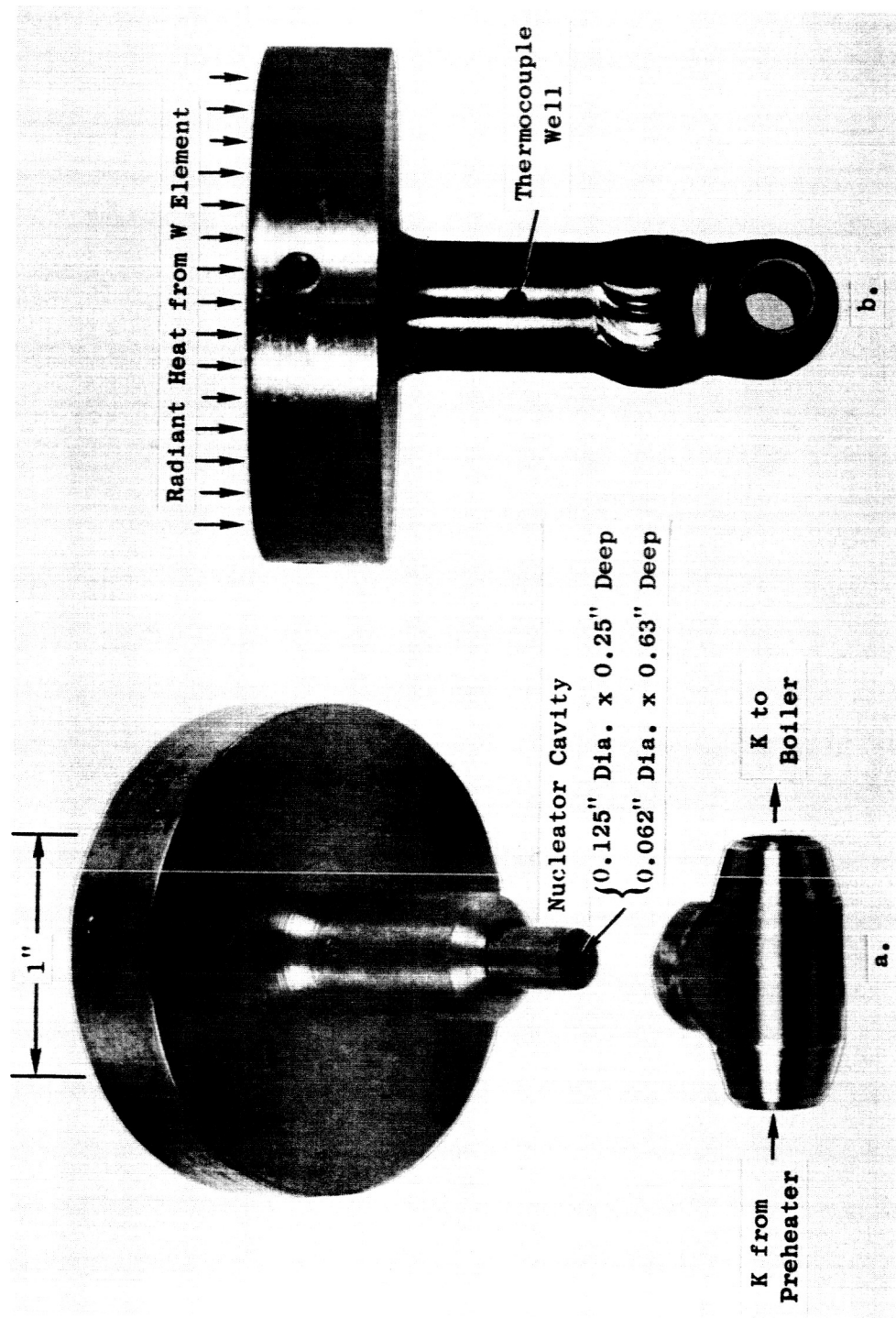
Figure 9. Vapor Nucleator (Cb-1Zr) for the Prototype Corrosion Loop a) Before and b) After Welding. a. (Orig. - C64122904) b. (Orig. - C64123003)

-25-
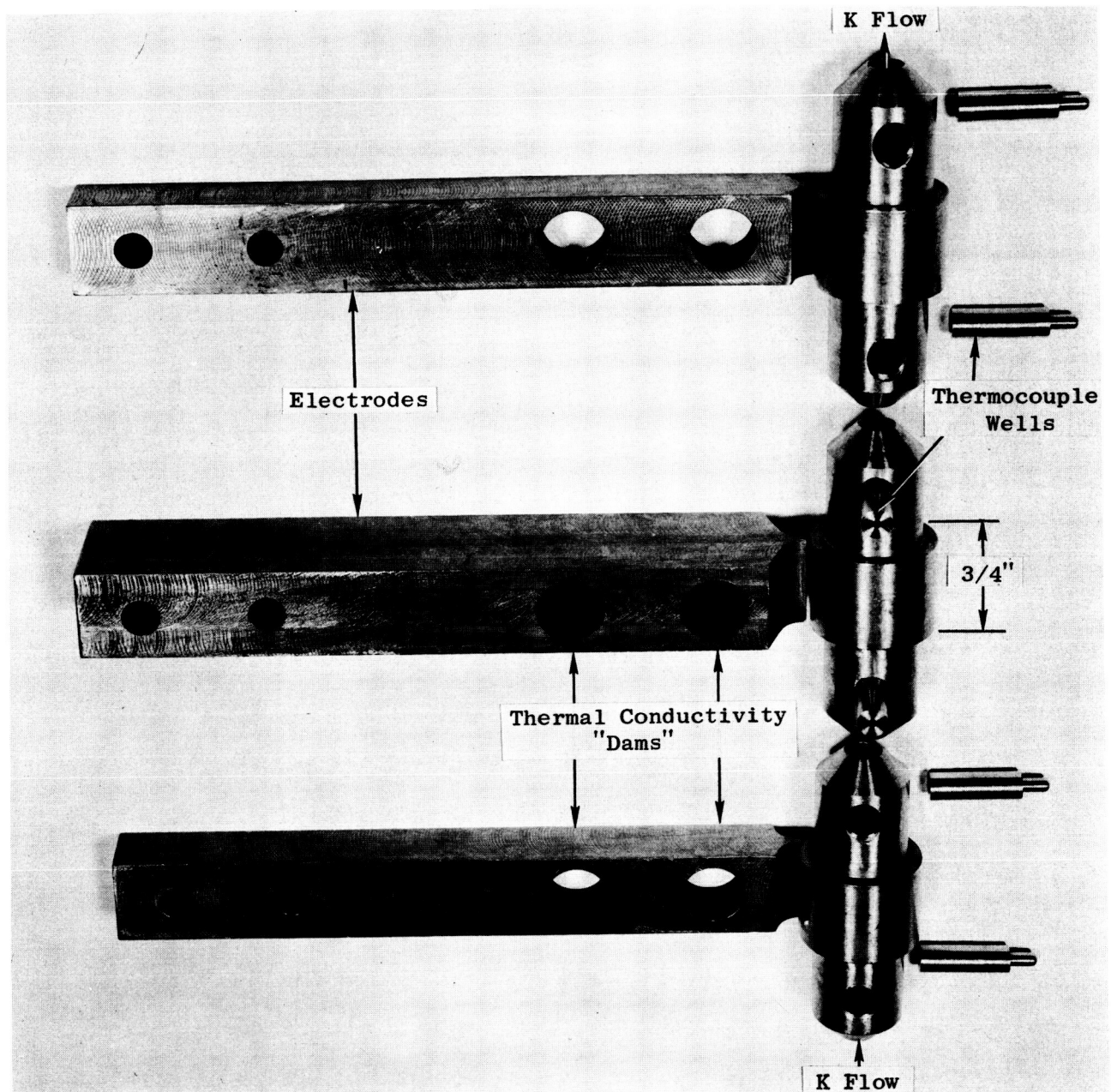
Figure 10. Potassium Preheater Coil Electrodes for the Prototype Corrosion Loop
Prior to Welding. (Orig. - C64122911)
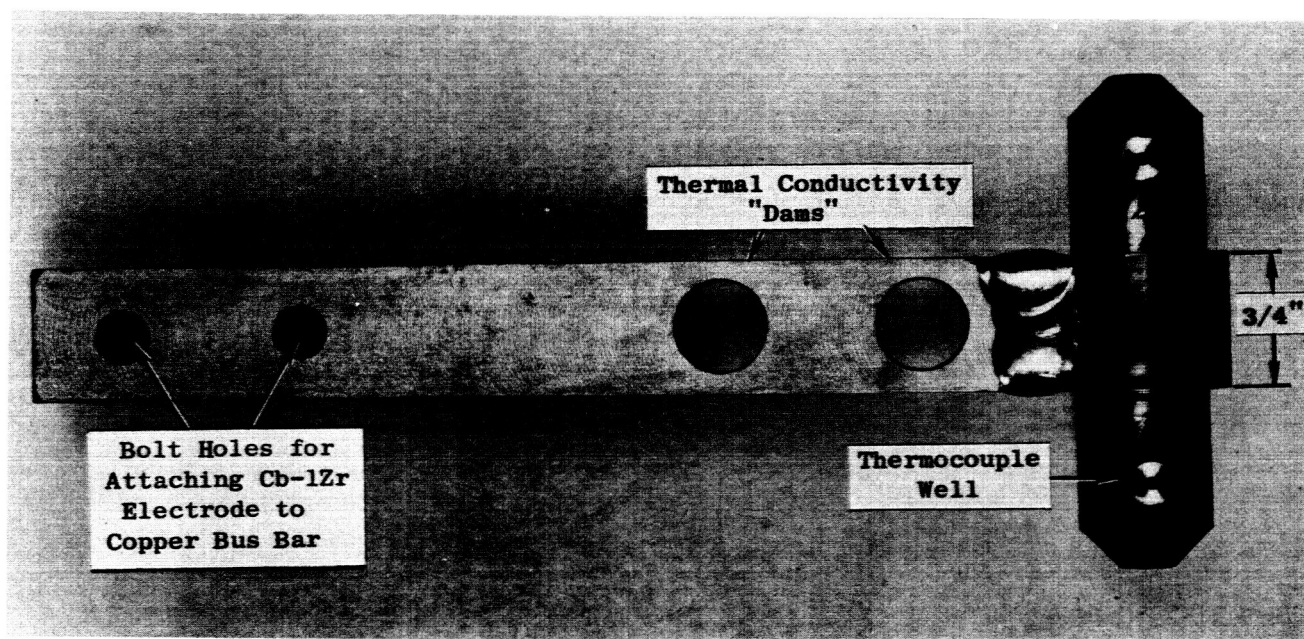
Figure 11. Potassium Preheater Coil Electrode for the Prototype Corrosion Loop
After Welding.   (Orig. - C64123004)
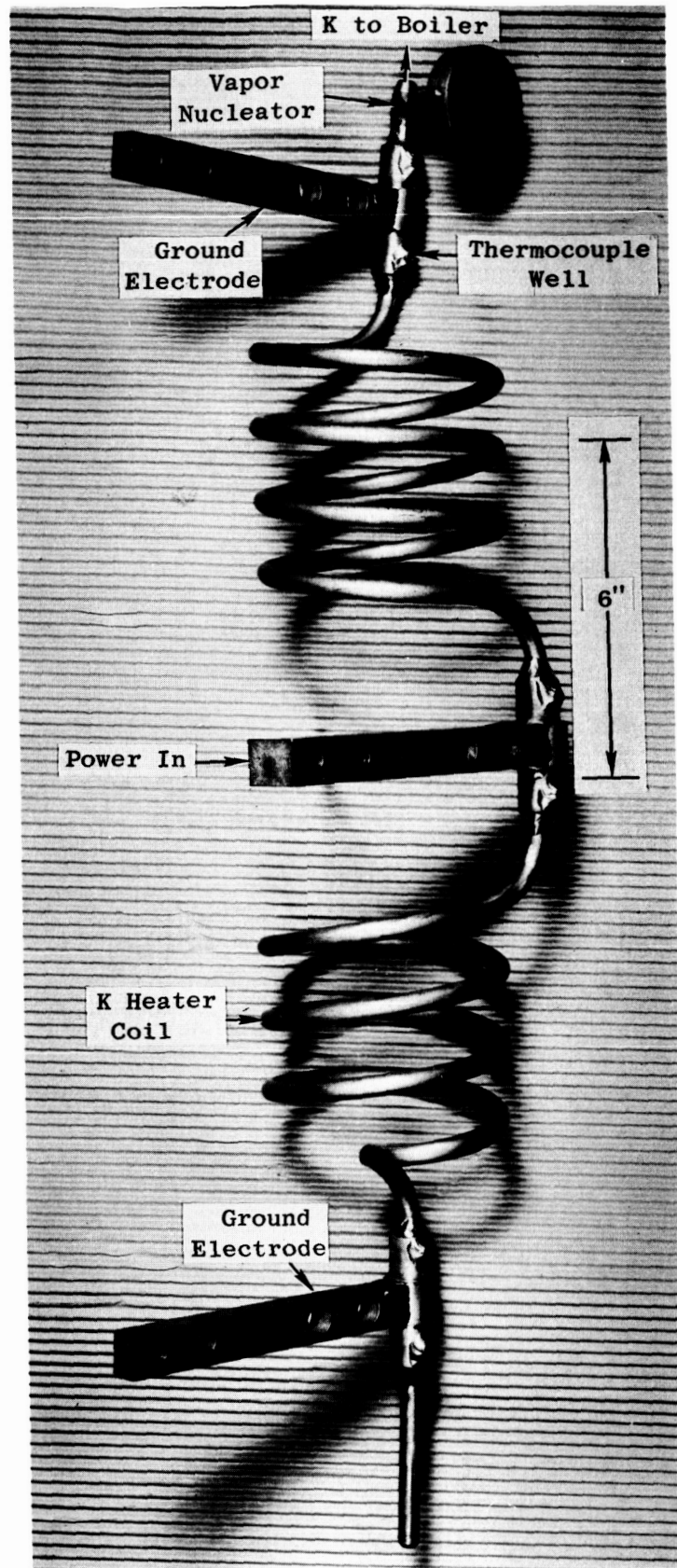
Figure 12. Potassium Preheater Coil-Vapor Nucleator Subassembly for the Prototype
Corrosion Loop. (Orig. - C65010631)

produced without difficulty. In each case, the outer wrapper was heated to
500°F in air and the finned duct was immersed in liquid nitrogen to facili-
tate the shrink fit. The closure welds were made following standard
procedures.

7. Stress Diaphragm Pressure Transducer

The components of the stressed diaphragm pressure transducer are
shown in Figure 13. During this report period, the tungsten arc welding
required for the body assembly was completed. A process tube (not shown in
Figure 13) was welded to the reducer and the assembly was post-weld annealed
per Specification SPPS-3C. The electron beam welding of the diaphragm
assembly will be completed during the next report period.

8. Other Components

The Cb-1Zr alloy components for the condenser subassembly were
received during this report period. In addition, stainless steel components
were received which will be used to fabricate a mock-up of the condenser.
This stainless steel mock-up will be used to establish techniques for appli-
cation of a high emittance coating on the condenser fins.

In preparation for the post-weld annealing of major loop sub-
assemblies, six welded test samples were annealed in the DuPont Company
vacuum furnace for 2 hours at 2200°F ± 50°F. These samples were unrapped
and, therefore, represent the worst condition for potential contamination.
The pressure at the start of the 2-hour anneal was 7×10^{-5} torr and
decreased to 5.5×10^{-5} torr at the end of the 2-hour anneal. Preliminary
chemical analyses of one sample indicated negligible contamination during
the furnace anneal as indicated in Table IV. Additional chemical analyses
and bend tests will be used to qualify this furnace per Specification SPPS-3C.

D. Prototype Corrosion Loop Components

1. Slack Diaphragm Pressure Transducers

The metallic impurity content of the NaK sample taken by Taylor[*]
during the filling of the pressure transducers was determined by Nuclear
Materials and Equipment Corporation with the following results as ppm of
chloride: Fe <5, B <5, Co <5, Mn <1, Al <5, Mg <1, Sn <5, Cu <1,
Pb <5, Cr <5, Si <10, Ti <5, Ni <5, Mo <5, V <5, Be <1, Ag <1, Zr <1,
Ba <2, Ca <1. The analysis of this NaK for oxygen by the amalgamation
method produced values of 3.3 and 4.3 ppm oxygen. Thus, it appears that the
purity of the NaK was maintained during the filling operation.

2. Stress Diaphragm Pressure Transducer

Purchase of a linear voltage differential transformer (LVDT) coil
for the Prototype transducer continued to be a problem. The vendor Consolidated
Controls Corporation) would not guarantee leak tightness of a standard coil

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[*]   Taylor Instrument Company, Rochester, New York.

-29-
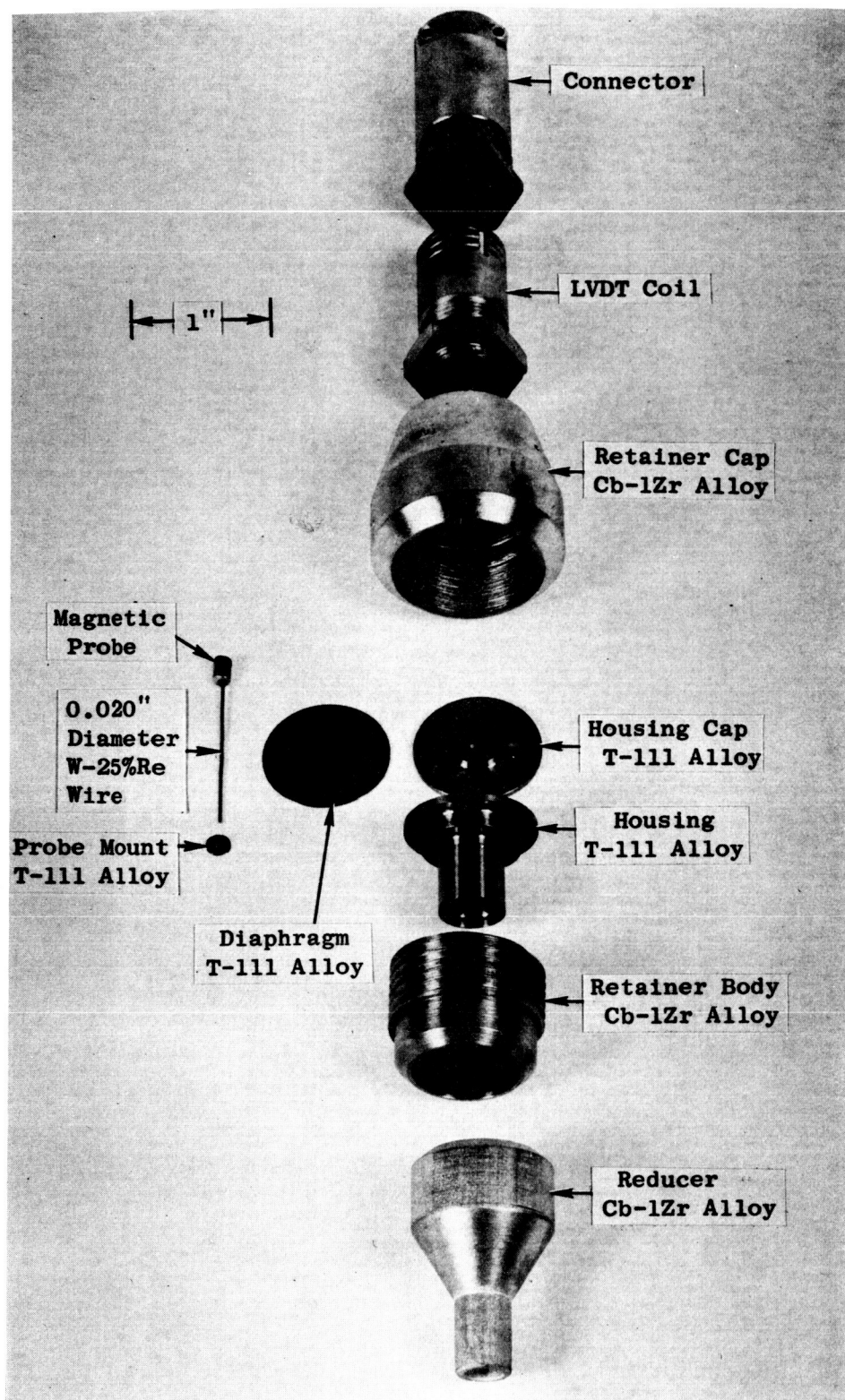
Figure 13. Stressed Diaphragm Transducer Assembly for the Prototype Corrosion Loop. (Orig. – C64122910)

TABLE IV

CHEMICAL ANALYSES OF WELDED Cb-1Zr ALLOY

SAMPLES ANNEALED IN THE DuPONT COMPANY VACUUM FURNACE*

| Sample | Condition | Chemical Analysis, ppm | | | |
		O	N	H	C
1	Parent metal, as-received**	56	14	6	10
2***	Parent metal following 2200°F, 2-hour anneal	89	19	2	20
3***	Weld metal following 2200°F, 2-hour anneal (weld process control No. 29)	99	16	1	10

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* Brew Company Cold-Wall, Tantalum-Heater Vacuum Furnace; Useable Furnace Volume: 42-Inch x 12-Inch x 104-Inch.

** All Samples were 0.040-Inch Thick Sheet Identified by MCN No. 454.

*** Pressure at Start of 2-hour anneal: 7×10^{-5} torr.
Pressure at End of 2-hour anneal: 5.5×10^{-5} torr.

due to the fact that the header used to seal the end of the coil housing can be damaged during final welding operations. Two available coils of the same design were tested for leak tightness by exposing them to 100 psi helium for one day and placing them in a vacuum tight container which was connected to a mass spectrometer leak detector. Both coils showed significant leaks and it is assumed that any new coil would also have a relatively large leak. Since it is not possible to get a leak tight coil without major redesign of the coil assembly, the coil to be used in the Prototype Corrosion Loop will be purchased with a 0.062-inch diameter hole drilled through the coil housing, and the assembly will be baked out in a vacuum for 100 hours at 750°F prior to loop installation.

3. Flow Test of the Metering Valve

The operating characteristics of the metering valve for the potassium circuit of the Prototype Corrosion Loop were experimentally determined in room temperature flow tests using high-purity nitrogen gas. The metering valve is a modified Hoke, Mfg. Company Model CB-442 bellows sealed valve with a 5/32-inch orifice and 3/8-inch diameter process tubes. All parts in contact with the liquid metal are fabricated from Cb-1Zr except for the molybdenum alloy Mo-TZM plug shown in Figure 14.

The manually operated valve has a maximum plug lift of 0.125 inch and is operated by a threaded stem in a ball bearing screw nut having a pitch of 0.125 inch. The lift off is equal to 0.001 inch for 3 degrees of rotation of the valve stem. Therefore, the first 30-40° (0.010-0.015 inch) of rotation is required to lift the plug sufficiently to raise the cylindrical section above the taper from the orifice. The 75° included angle tapered section is essentially the control surface. The remaining 95° (0.033 inch) of rotation increases the gap between the base of the plug and the seat until the flow area of the gap is approximately equal to the area of the 5/32-inch orifice.

The results of the flow test on the metering valve are shown in Figure 15 for nitrogen gas with a pressure loss of 1, 2, and 4 psi with an atmospheric discharge. The metering zone can be seen to extend from approximately 30 to 230° of rotation.

Flow coefficients based on the flow test were computed for the full range of plug lift off as a function of rotation. The flow coefficient computed for gases is essentially equivalent to liquid having viscosities equal to or less than water. The pressure drop as a function of rotation was then computed for potassium at the Prototype Loop test condition of 40 lbs/hr at 800°F and is shown in Figure 16.

These results indicate that control of potassium flow in the secondary circuit can be achieved in the pressure loss range of 1 to 50 psi, however, the control in the 10 to 50 psi range will be very sensitive to small changes in the plug position.

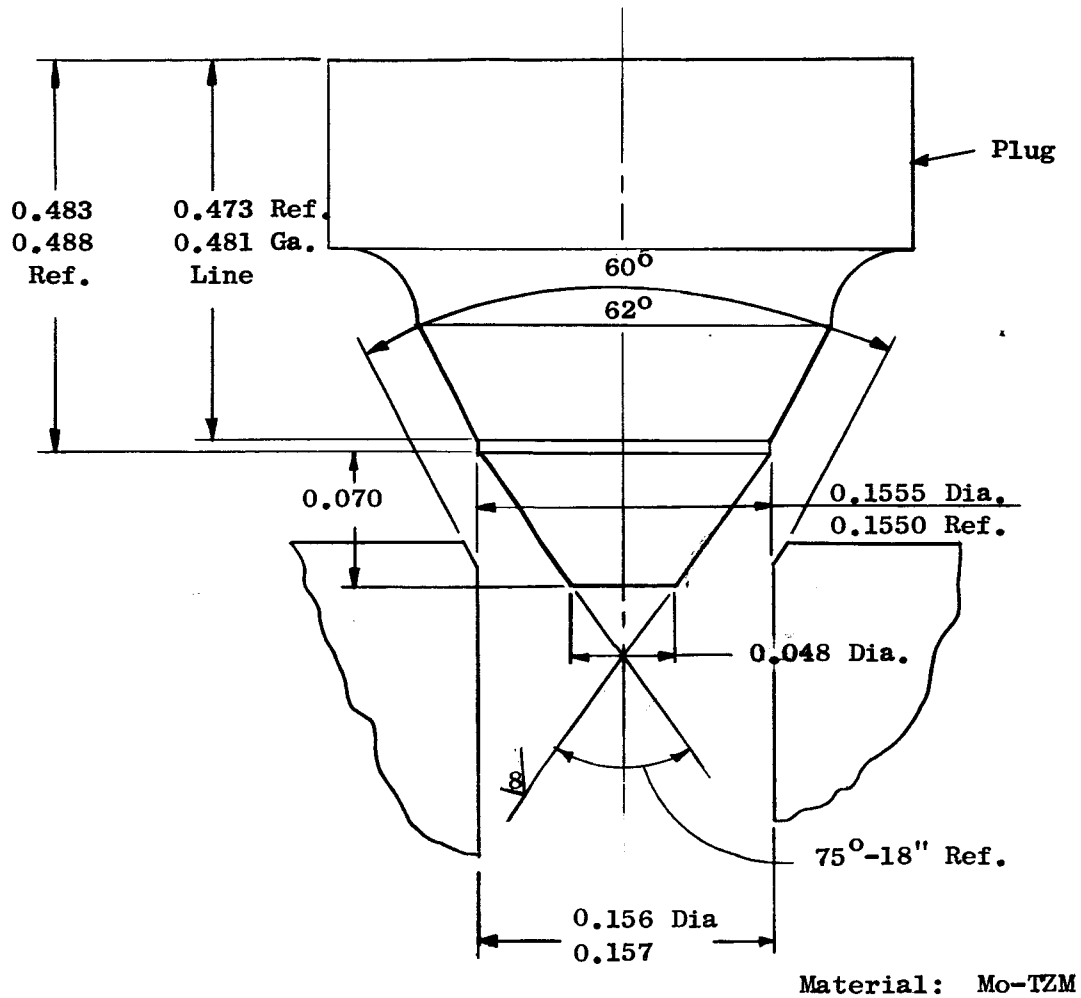Figure 14.   Detail of Metering Valve Plug Body Bore and Port
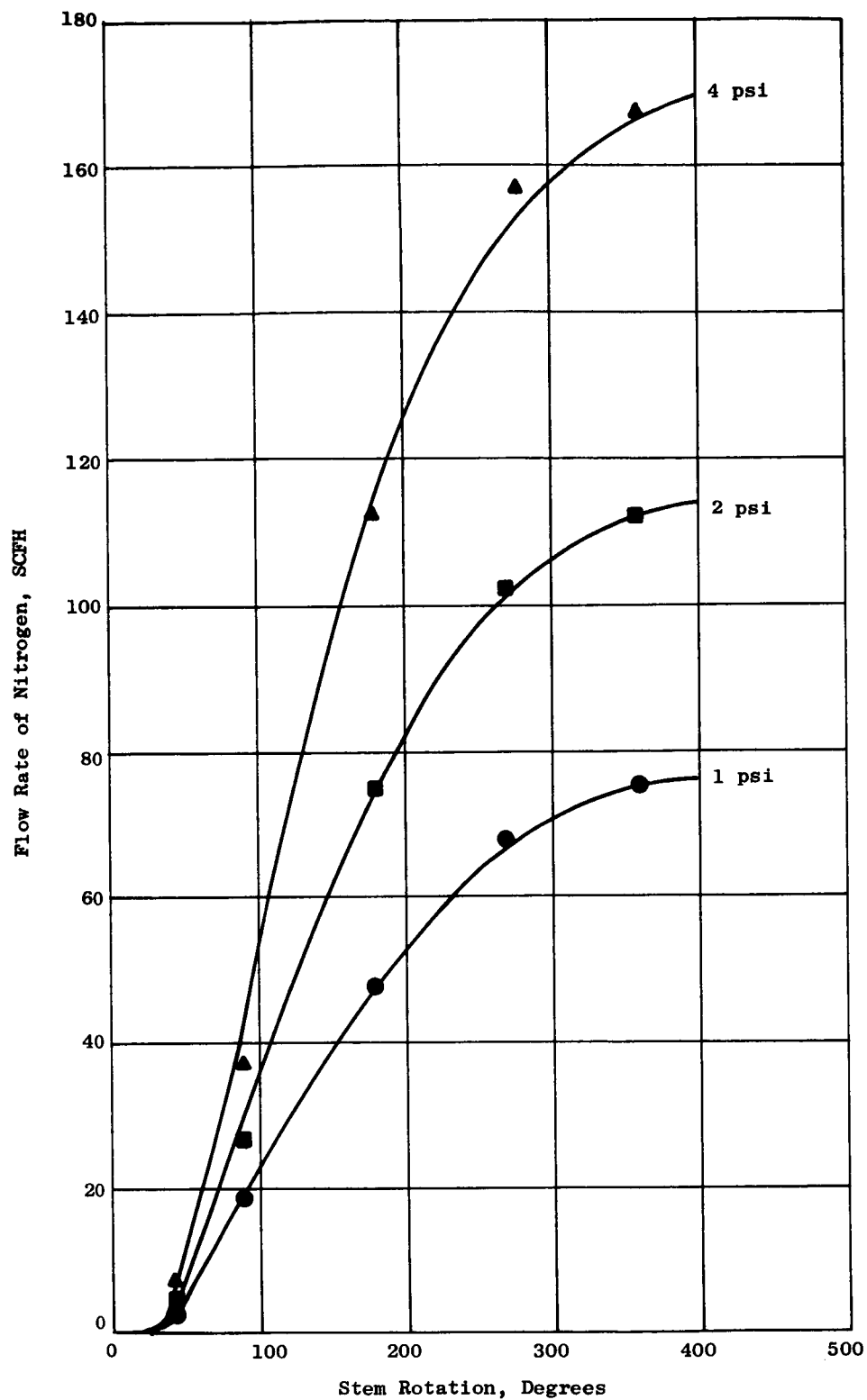for the Prototype Corrosion Loop.

Figure 15. Flow Rate for the Prototype Corrosion Loop Metering Valve as a Function of Stem Rotation for 1, 2 and 4 psi Pressure Drop Across the Valve.
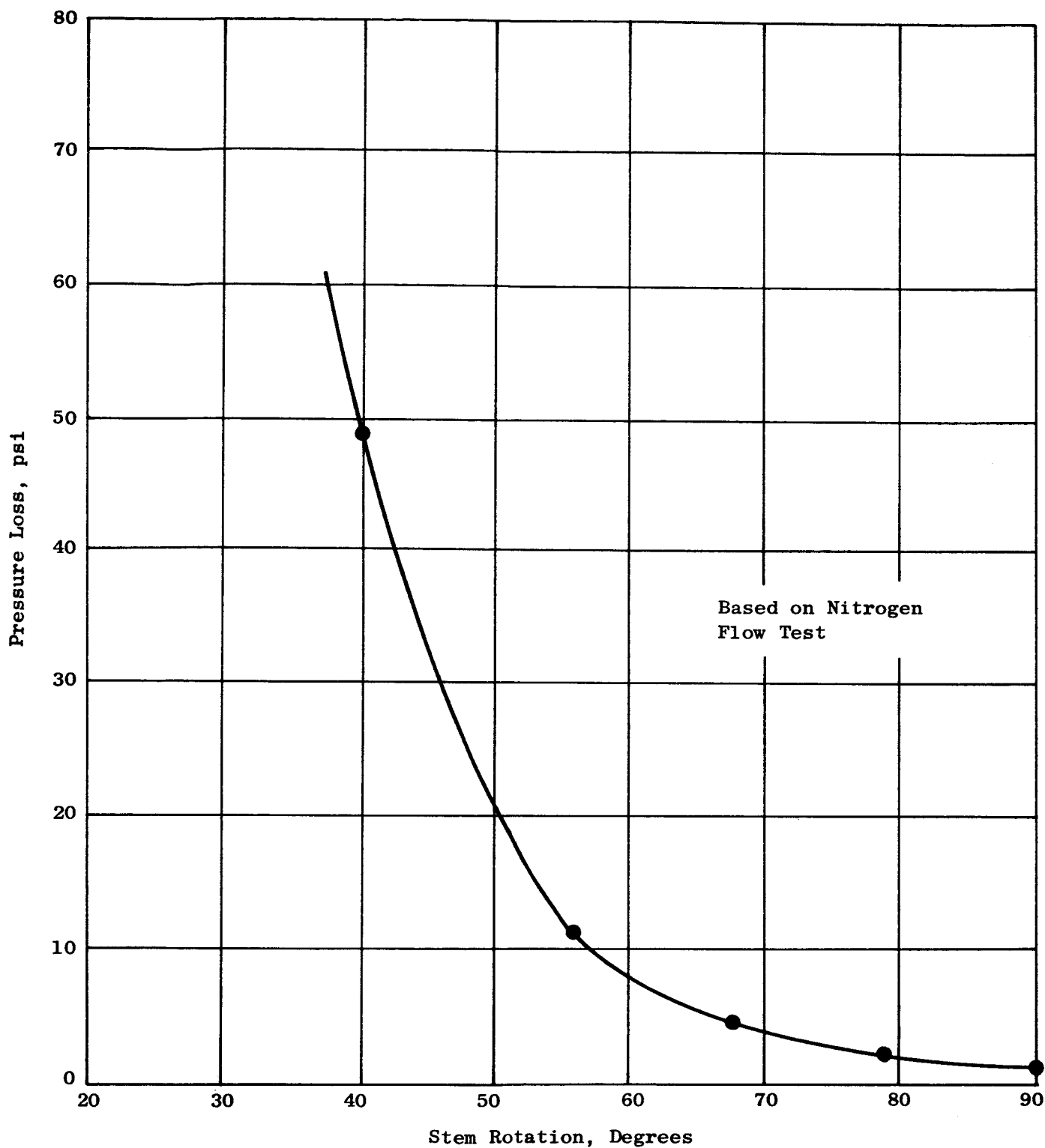
Figure 16. Prototype Corrosion Loop Metering Valve Pressure Loss as a Function of Stem Rotation for a Potassium Flow Rate of 40 Pounds Per Hour at 800°F.

4. Condenser Fin Coating Evaluation Test

As reported in a previous progress report[3], the original helical-type condenser was replaced with a finned straight tube condenser. This change was made to increase the entrance vapor velocity to 400 ft/sec in order to be in the range of interest for advanced space condensers. Subsequent emittance tests[4] on several types of grit-blasted Cb-1Zr alloy specimens indicated that emittance values of approximately 0.4 could be achieved. In order to increase the heat rejection capability of the 0.25-inch thick x 4-inch wide x 60-inch long fin, the NASA contract manager suggested that a high emittance coating of iron titanate (Fe_2TiO_5) on the Cb-1Zr alloy fin be considered for this application. A test program to evaluate this coating and others for Cb-1Zr is currently in progress at Pratt & Whitney Aircraft under NASA sponsorship. A recent report summarizes the current status of this program[5]. A total hemispherical emittance of 0.85-0.87 has been measured for an iron titanate coating on Cb-1Zr tubing which has operated for 1,950 hours at 1700°F in a vacuum of 10^{-8} torr. Pratt & Whitney has also determined that the 4-mil thick iron titanate coating will withstand rapid thermal cycling with no detectable deterioration in coating integrity.

The test which will be described below has been conducted by General Electric, in order to determine the thermal cycling characteristics of the iron titanate coating under test conditions quite similar to those which will exist in the Prototype Loop. Test specimens for this study were coated by R. Emanuelson of Pratt & Whitney. One specimen was grit blasted with alumina and the other with silicon carbide prior to the applications of the 3.5-mil layer of iron titanate by plasma-arc spraying using a non-oxidizing gas to form the plasma.

The test equipment consisted of an electrical resistance, radiation type furnace with a thermal capacity of 1.5 KW. All thermal insulation consisted of multiple layers of tantalum foil separated by a 0.020-inch diameter wire. The heater element was a single helical coil fabricated from 0.050-inch diameter tungsten wire. Two Cb-1Zr specimens, 0.25-inch thick x 2-inch wide x 2.5-inch long, were inserted into the furnace opposite the tungsten heating element as shown in Figure 17. Approximately 0.5 inch of the fin was left uncoated and this portion of the specimen was inserted into the furnace. The remaining 2-inch x 2-inch section of the fin projected out from the furnace and was held in position by small tantalum tabs which were spot welded to the furnace. The entire furnace with specimens was mounted

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[3] *Potassium Corrosion Test Loop Development*, Quarterly Progress Report No. 4 for Period Ending July 15, 1964, NASA Contract NAS 3-2547, NASA-CR-54167, p. 49.

[4] Ibid., p. 52.

[5] *Semi-annual Progress Report, Determination of the Emissivity of Materials*, Report Period: May 14-Nov. 15, 1964, R. C. Emanuelson, Pratt & Whitney Aircraft, NASA Contract NAS 3-4174, NASA-CR-54268.
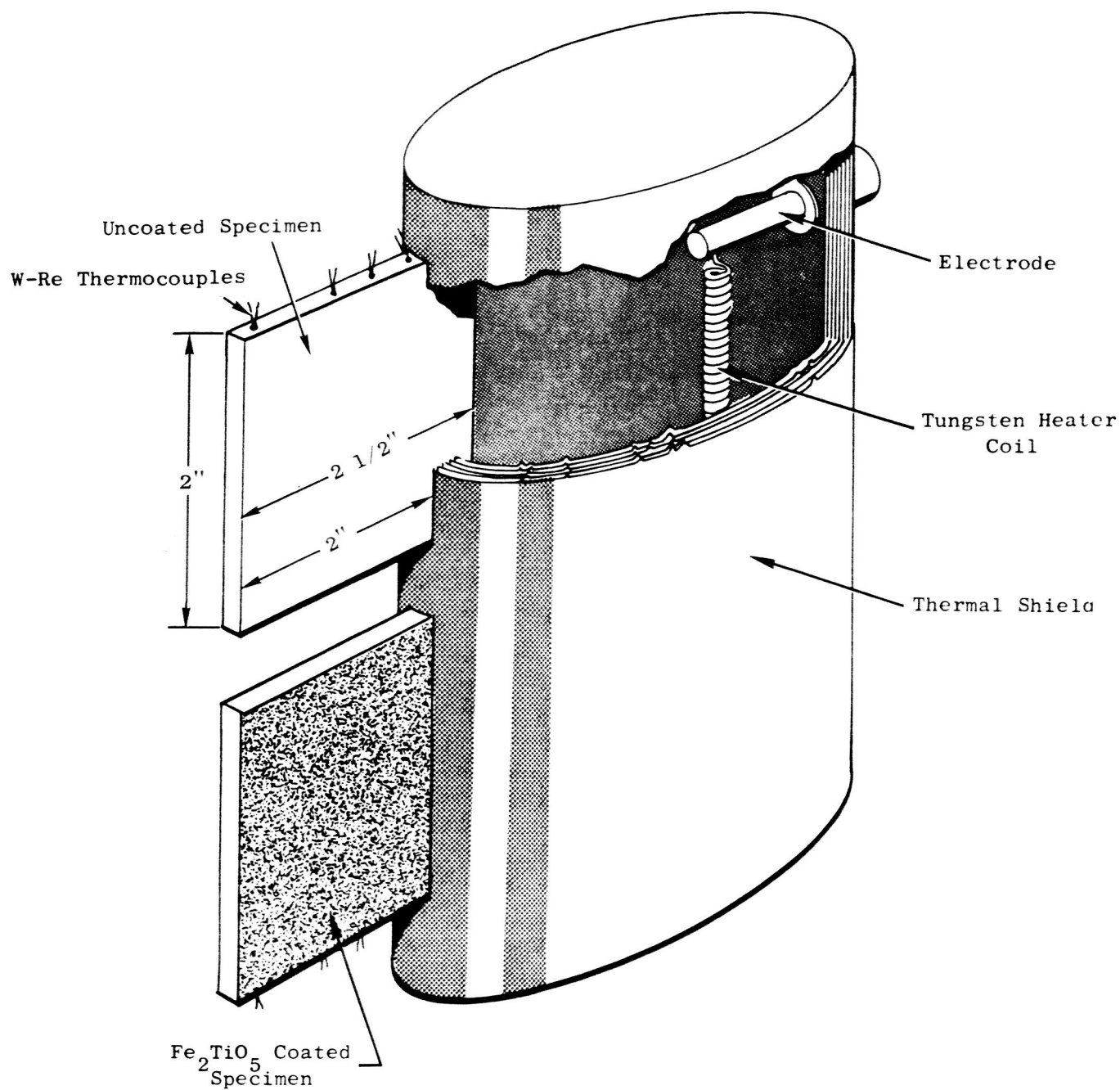
Figure 17. Test Configuration Used in the Evaluation of the High-Emittance, Fe_2TiO_5 Coating on Cb-1Zr Alloy Specimens. Uncoated, Grit-Blasted Specimen Included for the Purpose of Comparison. Specimens Radiated Heat to the Water Cooled Walls of a 10^{-8} Torr Vacuum Chamber.

in a 24-inch diameter ultra high vacuum system capable of 1×10^{-9} torr operation. The chamber walls were water-cooled to remove radiation heat loads from the specimens and furnace.

The test configuration illustrated in Figure 17 shows an uncoated and a coated specimen. For the first 236 hours of the test the top speci-ment was an iron titanate coated specimen which was grit blasted with sili-con carbide prior to application of the coating. After 236 hours of testing, this specimen was replaced with the uncoated grit-blasted specimen as shown in Figure 18.

Longitudinal temperature gradients in the specimen were measured by five W-3%Re/W-25%Re thermocouples attached to the edge of each specimen. These thermocouples were also used to obtain cooling rates during the thermal transient portion of the cycle;

The two coated samples differed only in the pre-coating surface roughening process. The bottom specimen was prepared by grit blasting with alumina grit and the top specimen was grit blasted with SiC grit.

Following the initial heating of the specimens to a maximum coated region temperature of 1500°F, the specimens were held at temperature for 77 hours. At this time the daily thermal cycling of the specimens was initiated. A test cycle consisted of the following: turning off the elec-trical power to the furnace in approximately one minute, allowing the speci-mens to cool by radiation to the chamber walls to a temperature of 150°F and then reheating the specimens to a temperature of 1500°F (temperature of hottest coated region) over a one-hour period. Seven thermal cycles of this type were completed in a total of 236 hours of test operation. The test was interrupted after 236 hours of test operation and seven thermal cycles to replace the silicon carbide grit blasted, iron titanate coated specimen with an uncoated, grit-blasted specimen.

During the 236 hours of test operation essentially no differences were detected in the temperature profiles of the two coated specimens as illustrated in Figure 19. No changes occurred with time or following cycling. This would suggest that there were no changes in coating integrity or emittance. Visual examination of the two specimens following opening of the test chamber revealed no evidence of spalling or coating deterioration.

As was mentioned above, an uncoated, alumina grit-blasted specimen was then substituted for the silicon carbide grit blasted and coated speci-men in order to obtain a comparison of the heat rejection properties of these two surface treatments.

The test was continued until a total of 1,000 hours of test time had been completed. During the 1,000-hour test, the alumina grit blasted, coated fin specimen was subjected to 25 thermal cycles at approximately 24-hour intervals during the first 750 hours of the test. Following the
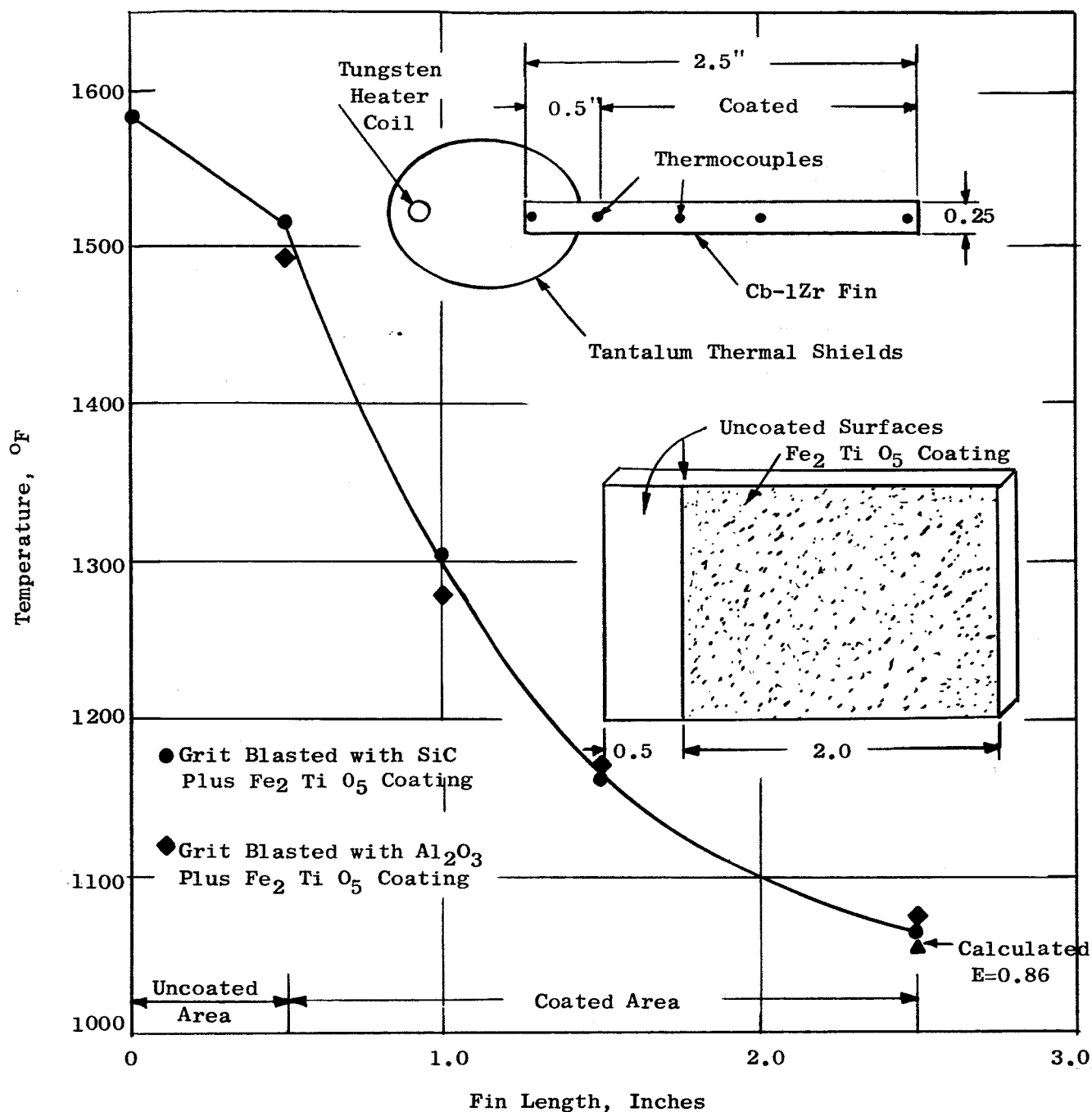
Figure 18. Steady State Temperature Profile of $Fe_2 Ti O_5$ Coated Cb-1Zr Alloy Specimens During Coating Evaluation Test. One Specimen Grit Blasted with SiC Prior to Coating with $Fe_2 Ti O_5$ and the other was Grit Blasted with Al_2O_3 Prior to Coating.
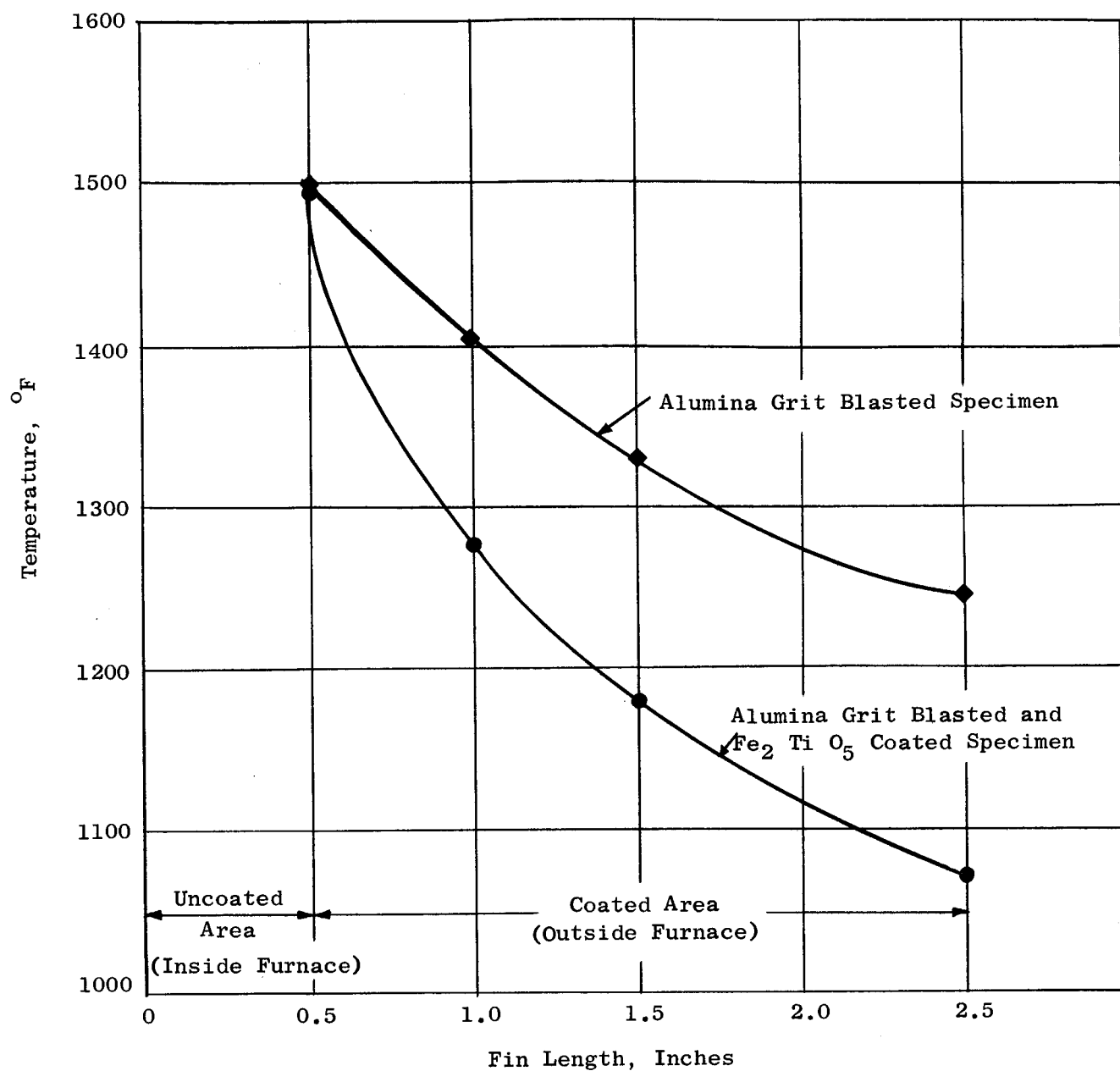
Figure 19. Steady State Temperature Profile of Coated and Uncoated Cb-1Zr Alloy
Fin Specimens During Coating Evaluation Test.

25th cycle, the specimen was held at temperature for an additional 250 hours and then subjected to one final cycle. A summary of the thermal history of the coated specimen is given below:

At temperature:	922 hours
Cooling to 150°F:	52 hours
Heating to 1500°F:	<u>26 hours</u>
TOTAL	1,000 hours

During the experiment, the pressure in the system (with the specimens at temperature) decreased from 9.0×10^{-9} torr at the start of the test to 9.5×10^{-10} torr at the completion of the test.

The temperature distributions along the coated and the grit blasted fin specimens are shown in Figure 19. The effectiveness of the coating treatment is quite evident, yielding a fin end temperature of 1070°F compared to a temperature of 1245°F for the grit blasted, uncoated specimen.

The end temperature of a Cb-1Zr fin specimen having the dimensions of the specimens used in this experiment, as a function of surface emittance was calculated using the relationships developed by S. Leiblein[6]. The curve developed is shown in Figure 20. The actual end temperature of the coated fin, 1070°F, and the actual emittance valve measured by Pratt & Whitney[7] for this coating at 1800°F on tubular specimens are also shown on this figure. It may be noted that the actual end temperature is within 10°F of the calculated value based on the measured emittance (0.85-0.87). The discrepancy, though small, would be even less if the outer edge (1/4-inch x 2-inch) of the specimen had been coated.

It may also be noted in Figure 20 that the effective emittance of the uncoated, grit-blasted specimen was approximately 0.3, which is considerably less than the value of 0.42 previously measured[8] on sheet specimens. Differences in the grit blasting treatment are probably responsible for this discrepancy.

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[6] Leiblein, S., Analysis of Temperature Distribution and Radiant Heat Transfer Along a Rectangular Fin of Constant Thickness, NASA Technical Note D-196, November, 1959.

[7] Semi-annual Progress Report, Determination of the Emissivity of Materials, Report Period: May 14-Nov. 15, 1964, R. C. Emanuelson, Pratt & Whitney Aircraft, NASA Contract NAS 3-4174, NASA-CR-54268.

[8] Potassium Corrosion Test Loop Development, Quarterly Progress Report No. 4 for Period Ending July 15, 1964, NASA Contract NAS 3-2547, NASA-CR-54167, p. 49.
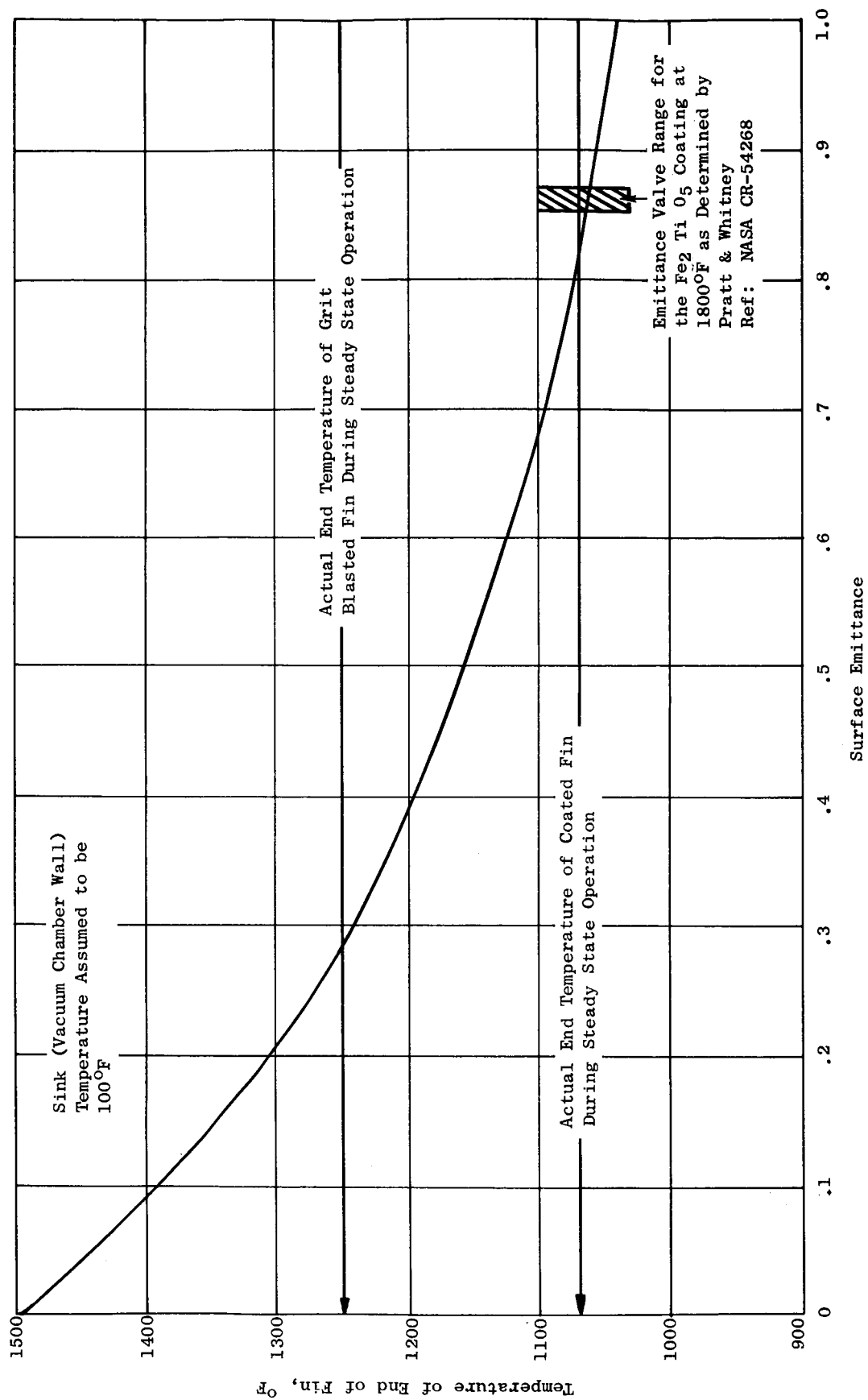
Figure 20. End Temperature of a 2-Inch x 2-Inch x 0.25-Inch Thick Cb-1Zr Fin with a Base Temperature of 1500°F as a Function of Surface Emittance.

A further comparison of the emittances of the specimens can be made from the cooling rates obtained during the thermal cycling phase of the test. The heat loss from the specimen can be expressed by:

$$dQ = \sigma \, \epsilon \, A \, (T_s^4 - T_w^4) \, dt$$

where

Q = Heat radiated per unit time

σ = Stefan-Boltzmann constant

ϵ = Hemispherical emittance

A = Surface area of fin

T_s = Absolute temperature of radiating surface

T_w = Absolute temperature of chamber wall

and

$$dQ = W \, C \, d \, T$$

where

W = Weight of fin

C = Heat capacity of Cb-1Zr

T = Temperature of fin

Equating the above equations we obtain:

$$\frac{dT}{dt} = \frac{\sigma \, \epsilon \, A}{W \, C} \, (T_s^4 - T_w^4)$$

For similar specimens differing only in their surface emittances, the ratio of the cooling rates, $\frac{dT}{dt}$, evaluated at the same surface temperature should

be equal to the ratio of the emittances. The cooling rate equation can be determined from Figure 21 where the specimen temperatures are shown as a function of time which can be expressed as:

$$\log T = A \log t + \log b \qquad t \geq 1$$

or

$$T = bt^{A} \qquad t \geq 1$$

differentiating and collecting terms, we obtain:

$$\frac{dT}{dt} = A b t^{A-1}$$

Substituting the experimental value from Figure 20, we obtain:

$$Fe_2TiO_5 \text{ coated specimen} \qquad \frac{dT}{dt} = \frac{322}{t^{1.22}} \qquad t \geq 1$$

$$\text{Uncoated specimen} \qquad \frac{dT}{dt} = \frac{494}{t^{1.25}} \qquad t \geq 1$$

Evaluating the cooling rates at 1000° and 800°F, we obtain with the aid of Figure 21, the following values:

Uncoated			Coated	
T	t	$\frac{dT}{dt}$	t	$\frac{dT}{dt}$
1000°F	3.5 min	104°F/min	1.05 min	303°F/min
800°F	6.3 min	50°F/min	2.0 min	138°F/min

The ratio of the cooling rates, $\dfrac{\frac{dT}{dt} \text{ uncoated}}{\frac{dT}{dt} \text{ coated}}$ is equal to 0.34 evaluated at 1000°F and 0.36 evaluated at 800°F which is in good agreement with the ratio of the emittance (0.35) of the specimens obtained in steady state testing. (Figure 20).

E. Refluxing Potassium Compatibility Tests

The two refluxing potassium capsule tests which are being conducted to determine the extent of mass transfer of Mo-TZM alloy tubular insert specimens in the condenser region of Cb-1Zr alloy capsules have been started. The test system has been described in previous progress reports[9,10].

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[9] Potassium Corrosion Test Loop Development, Quarterly Progress Report No. 3 for Period Ending April 15, 1964, NASA Contract NAS 3-2547, NASA-CR-54081, p. 37.

[10] Potassium Corrosion Test Loop Development, Quarterly Progress Report No. 5 for Period Ending October 15, 1964, NASA Contract NAS 3-2547, NASA-CR-54269, p. 28.
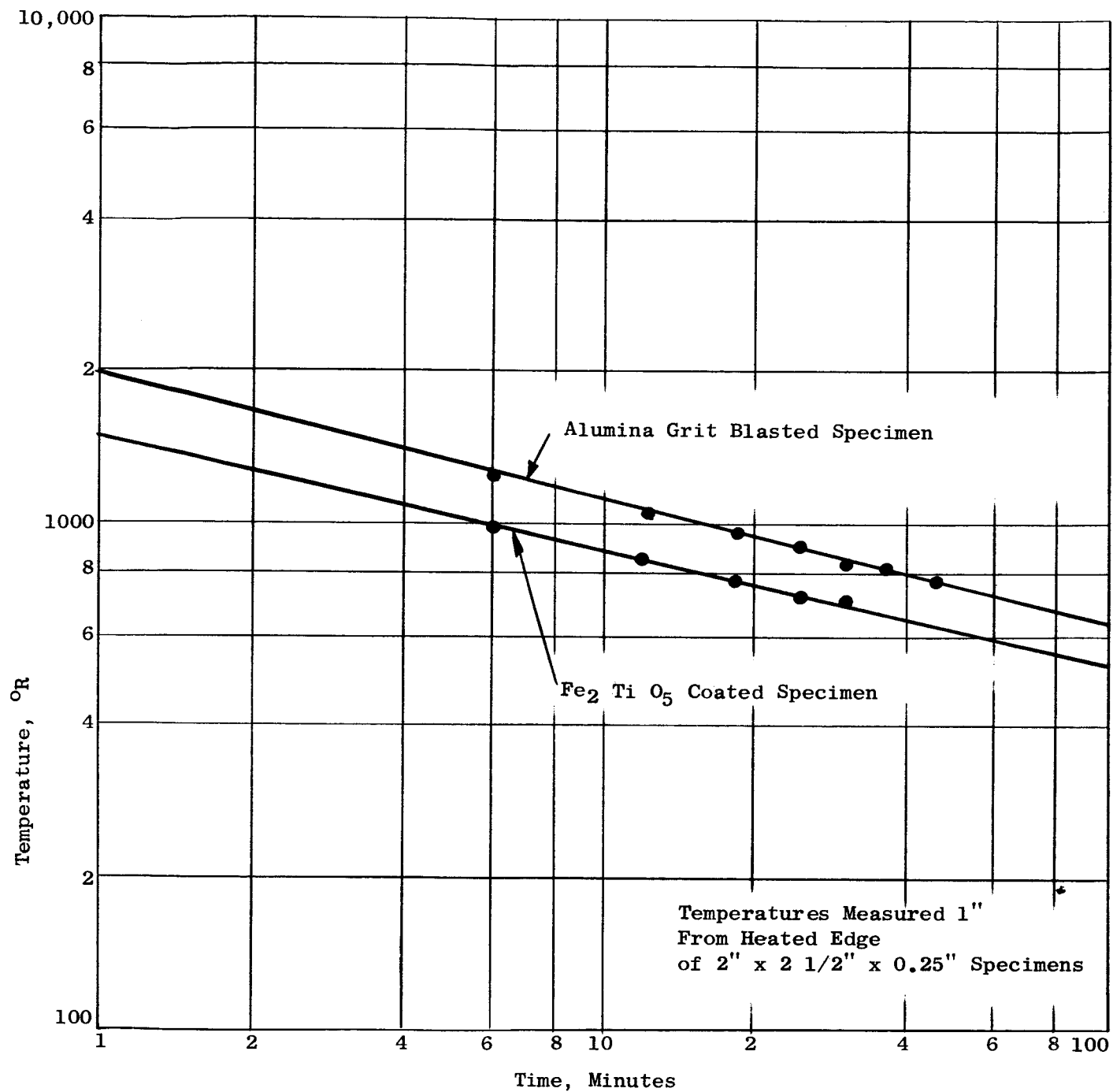
Figure 21.   Cooling Curves for the Coated and the Grit Blasted Cb-1Zr Fin
Specimens.

After placing the capsules in the test facility and instrumenting them with
W-3%Re/W-25%Re thermocouples, the ultra-high vacuum chamber was sealed and evac-
uated to a pressure of 50 microns of Hg using a molecular sieve trapped mechanical
pump. Using cryogenic sorbtion pumps, the pressure was reduced to 1.5 microns
of Hg at which time the getter-ion pump was started. The pressure prior to bake-
out was 2.0×10^{-8} torr. After a 14-hour bakeout at 400°F, the pressure decreased
to 2.2×10^{-9} torr. All pressures monitored in the ultra-high vacuum range were
obtained using a Bayard-Alpert ionization gauge located on the side of the chamber.

Power was applied to both heaters and the test temperature of 2000°F was
reached in 45 hours while the pressure was maintained at less than 1×10^{-6} torr.
Unstable boiling was encountered in the temperature range 1300° to 1700°F in both
capsules. The liquid potassium temperature was recorded as high as 148°F above
the vapor temperature during this unstable condition, and as low as 18°F above
the vapor temperature during moments of violent boiling. Two thermocouples, one
in the liquid zone and one on the external surface of the condensing zone of
Capsule #1, became inoperative after the unstable boiling.

The two capsules have completed 1,243 hours of testing at 2000°F. The pres-
sure in the vacuum chamber at this time is 1.0×10^{-8} torr. The temperatures of
the various regions of the two capsules are given in Table V.

The average condensing rates of potassium as determined by the heat measure-
ment technique previously described and using a value of 736.9 BTU/lb as the heat
of condensation[11] are given below:

    Capsule #1   19.8 lbs/ft^2/hr (0.160 gm/cm^2/min)

    Capsule #2   20.2 lbs/ft^2/hr (0.165 gm/cm^2/min)

Capsule #1 will be removed from the test chamber and evaluated after com-
pletion of 2,500 hours of operation. Capsule #2 will be tested for an additional
2,500 hours.

F.   Helium Analysis System

Due to the difficulty in obtaining reliable analyses for water with the mass
spectrometer[12], an electrolytic hygrometer system has been designed and constructed
for the determination of moisture within the welding chamber. Figure 22 shows a
schematic diagram of the system. Sample gas from the welding chamber is contin-
uously drawn through the electrolytic cell and the flowmeter transducer. The
electrolytic cell is used to determine the water vapor flow rate and the flowmeter

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[11] Ewing, C. T., Stone, J. P., Spann, J. R., Steinkuller, E. W., Williams, D. D.,
and Miller, R. R., "High Temperature Properties of Sodium and Potassium," NRL
Report 6128, August, 1964.

[12] Potassium Corrosion Test Loop Development, Quarterly Progress Report No. 4
for Period Ending July 15, 1964, NASA Contract NAS 3-2547, NASA-CR-54167,
p. 62.

TABLE V

TEMPERATURE OF CAPSULES #1 AND #2

Location	Mean Temp. °F	Mean Deviation °F
Capsule #1		
Thermocouple well in condensing region	1991	± 5.0
Brightness optical pyrometer in condensing region opposite thermocouple well[1]	1908	± 3.4
Capsule #2		
Thermocouple well in liquid region	2006	± 3.6
Thermocouple well in condensing region	2000	± 3.8
Thermocouple on external surface of capsule opposite thermocouple well	1993	± 4.6
Brightness optical pyrometer in condensing region opposite thermocouple well[1]	1904	± 1.0

----

(1) Values Lack Correction for Emissivity of Surface.

(1) Inlet Shutoff Valve - Whitey Ball Valve Type 43S4-316

(2) Electrolytic Hygrometer Cell - Beckman Part No. 76285

(3) Mass Flowmeter Transducer - Hastings-Raydist Type F-100

(4) Metering Valve - Nupro Type SS2S

(5) Vacuum Gauge - USG Type 19931-1; 0-30 in. Hg vac.

(6) Outlet Shutoff Valve - Hoke Toggle Type 1252

(7) Vacuum Pump - Welch No. 1399B

(8) Hygrometer Cell Milliammeter - Weston
    Model 911 (Multirange)
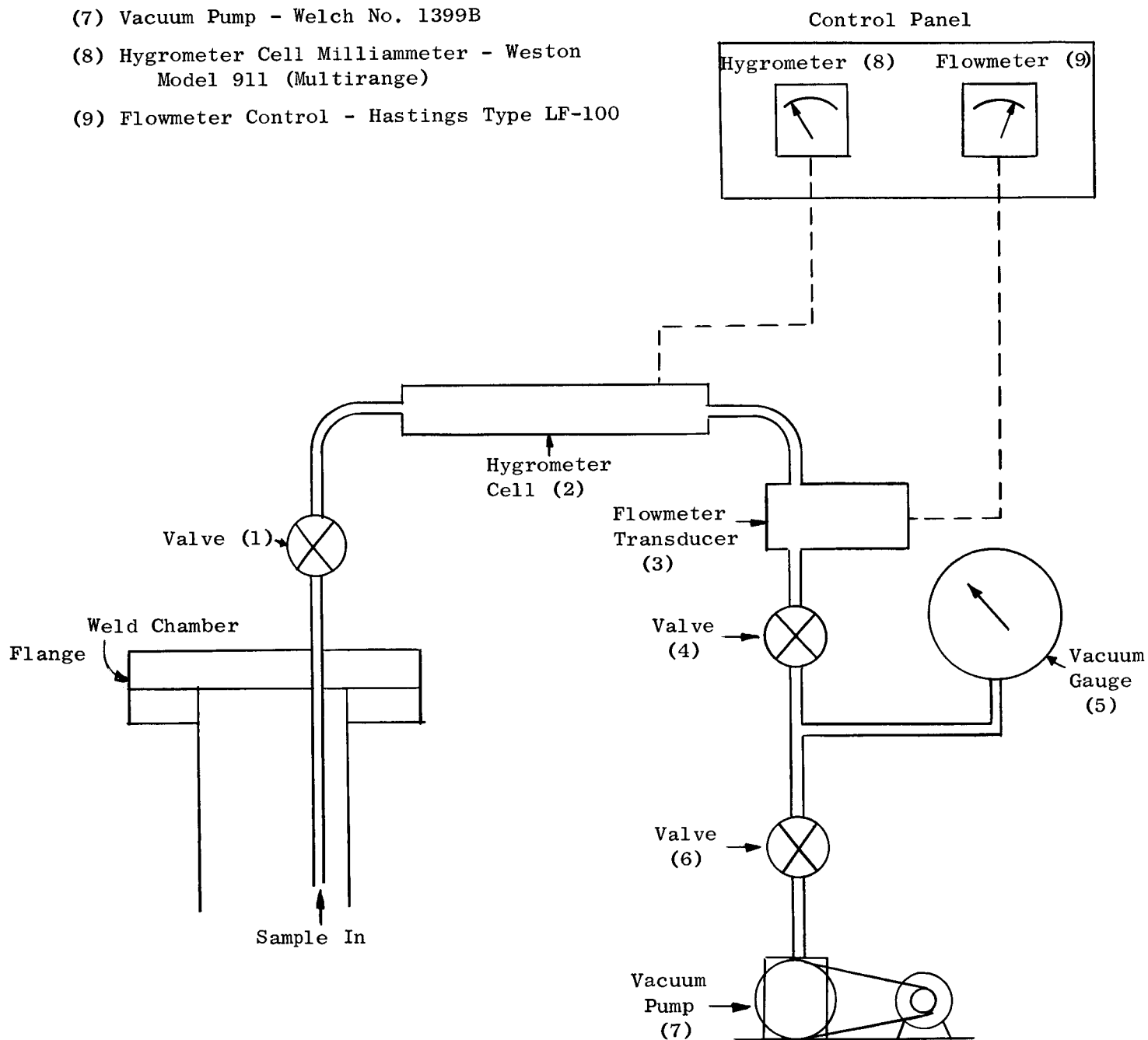
(9) Flowmeter Control - Hastings Type LF-100

Figure 22. Schematic of Electrolytic Hygrometer System for Moisture Measurement
in Welding Chamber.

measures the total gas flow. To decrease the response time of the system, the length of sample line and the number of fittings between the cell and the welding chamber have been minimized. With the welding chamber helium pressure automatically regulated and critical flow through the metering valve, no additional flow regulation has been found to be necessary. The electrical circuit for the cell consists of a 67.5-volt battery connected in series with the cell, a 10,000 ohms current limiting resistor, the milliammeter, and a switch. The multi-range milliammeter gives full scale indication for 0.1, 0.3, 1.0, 3.0, 10.0, 30.0 or 100 milliamps.

The operation of the hygrometer is based on Faraday's law which, when applied to the electrolysis of water, states that for each Faraday (96,500 coulombs), 9.008 grams of water (one gram equivalent weight) dissociate into hydrogen and oxygen gas. If water vapor is being electrolyzed at a constant rate, this rate is $\frac{9.008}{96,500}$ = 9.34 x 10^{-5} gm/sec of water vapor/ampere of electrolyzing current. This value may be converted to standard volume units to obtain 0.1266 std cc/ sec per ampere of electrolyzing current when standard conditions are taken as 25°C and 760 torr pressure. Using this factor, it may readily be shown that moisture content (ppm by vol) =

$$\frac{7.60 \text{ x electrolyzing current (microamperes).}}{\text{total flow rate (std cc/min)}}$$

It has been found quite convenient to set the total flow rate at 76.0 std/ cc/min so that the moisture content in ppm by volume is just 0.1 times the cell current in microamperes.

The Hastings-Raydist type LF-100 mass flowmeter reads directly 0-100 cc/ min of air at standard conditions. Calibration was performed here for helium using a "bubble flowmeter", and several calibration points were also obtained using a wet test meter. All calibration points have been corrected to standard conditions (i.e., 25°C and 760 torr pressure). The results of this calibration are shown in Figure 23. The manufacturer of the flowmeter states that the instrument is accurate within 2% at pressures from 0.01 psia to 250 psia and temperatures to 200°F. The calibration points of Figure 23 were obtained with helium pressures at the transducer between atmospheric and 10 psig and no particular trend with pressure was noted.

The minimum current through the cell that has been obtained to date is 9 microamps, corresponding to a moisture indication under normal flow conditions of about 1 ppm. Since this value has been obtained under both no flow and normal flow conditions, the present tendency is to consider this a "background" current which could arise from 1) residual resistance of the cell, 2) the fact that the flow system is still not completely dry, or 3) leakage of atmospheric moisture into the system. Further investigation of the source of this background current is planned.

Although only a limited amount of data has thus far been obtained on the quality of moisture in the welding chamber and the change of moisture content under various conditions, the following effects have been noted:
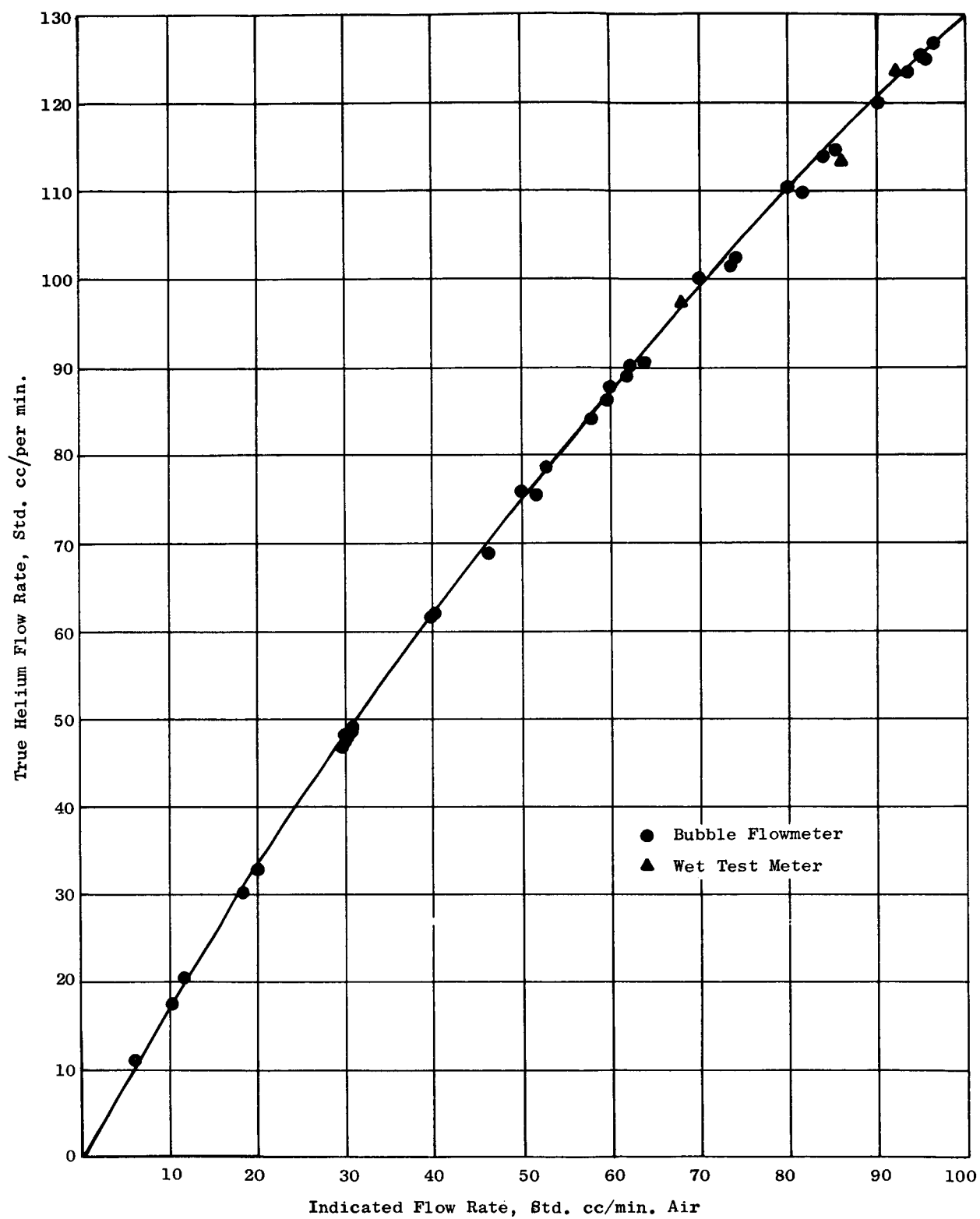
Figure 23. Calibration of Flowmeter for Helium.

1. Upon initial filling of the chamber, the moisture content is essentially zero and will remain below the 1 ppm level for a period of about an hour if no heat is applied to the interior of the chamber.

2. Heat applied to the chamber, either by welding inside or by heating the chamber walls by passing hot water through the jacket, results in an increase in moisture concentration.

3. Overnight "bakeout" of the chamber with hot water (approximately 120°F) results in a decrease in the quantity of moisture observed upon subsequent filling of the chamber.

These observations indicate that there is both a source and a sink for water vapor within the chamber and that the net change in moisture content depends on which of the two processes predominates. The behavior of the water vapor content within the welding chamber and the tentative conclusions reached here are quite similar to those obtained at Westinghouse Astronuclear Lab[13].

_____

13
Lessman,, G. G. and Stoner, D. R., Determination of the Weldability and Elevated Temperature Stability of Refractory Metal Alloys, Quarterly Report No. 3 for Period Dec. 21, 1963 to Mar. 21, 1964. Contract NAS 3-2540, Westinghouse Electric Corporation, Astronuclear Laboratory.

IV   FUTURE WORK

A.   Fabrication of the Prototype Corrosion Test Loop will be completed and instrumentation of the loop will begin.

B.   The fabrication of the alkali metal purification and handling systems for the Prototype Corrosion Test Loop will be completed and purification of the potassium by hot gettering and distillation initiated.

C.   All instrumentation tasks on the Prototype Corrosion Test Loop which can be accomplished prior to completion of the fabrication of the loop will be finished.

D.   Refluxing Potassium Capsule #1 will be taken off test when 2,500 hours of operation has been completed on March 3. Capsule #2 will be tested an additional 2,500 hours.

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